Formerly Utilized Sites
Remedial Action Program
(FUSRAP)

Maywood Chemical Company Superfund Site

ADMINISTRATIVE RECORD

Operable Unit 2 - Groundwater

Document Number

GW-015

US Army Corps
of Engineers®
New York District

FUSRAP
Maywood Superfund Site

CDQMP Volume 2, Field Sampling Plan

Formerly Utilized Sites Remedial Action Program
Maywood Superfund Site

Prepared by:

Shaw Environmental, Inc.
100 West Hunter Avenue
Maywood, New Jersey 07607

Prepared for:

US Army Corps of Engineers®

Contract No. DACW41-99-D-9001

June 2009, Revision 2

CHEMICAL DATA QUALITY MANAGEMENT PLAN
VOLUME 2 – FIELD SAMPLING PLAN

FUSRAP MAYWOOD SUPERFUND SITE
MAYWOOD, NEW JERSEY

SITE-SPECIFIC ENVIRONMENTAL RESTORATION
CONTRACT No. DACW41-99-D-9001
TASK ORDER No. 0003
WAD 01 WBS 01

Submitted to:

Department of the Army
U.S. Army Engineer District, New York
Corps of Engineers
FUSRAP Project Office
26 Federal Plaza
New York, New York 10278

Department of the Army
U.S. Army Engineer District, Kansas City
Corps of Engineers
700 Federal Building
Kansas City, Missouri 64106

Submitted by:

Shaw Environmental, Inc.
100 West Hunter Avenue
Maywood, New Jersey 07607

June 2009
Revision 2

Volume 2

This page intentionally left blank.

TABLE OF CONTENTS

LIST OF TABLES

APPENDICES

ABBREVIATIONS AND ACRONYMS

°C	degrees Celsius
CDQMP	Chemical Data Quality Management Plan
CFR	Code of Federal Regulations
CO	Contracts Officer
COC	Chain of Custody
DCGL	Derived Concentration Guideline Level
DOT	Department of Transportation
DQCR	Daily Quality Control Report
DQO	Data Quality Objective
EDMS	Electronic Document Management System
EE/CA	Engineering Evaluation / Cost Analysis
EM	Engineering Manual
EMP	Environmental Monitoring Program
EPA	U.S. Environmental Protection Agency
FMSS	FUSRAP Maywood Superfund Site
FSP	Field Sampling Plan
FSS	Final Status Survey
FUSRAP	Formerly Utilized Sites Remedial Action Program
g	gram
GEPP	General Environmental Protection Plan
I-80W	Interstate 80 Westbound
IDW	investigation-derived waste
ISOCS®	In-Situ Object Counting System
L	liter
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MD	matrix duplicate
MHT&D	Materials Handling, Transport, and Disposal
MISS	Maywood Interim Storage Site
mL	milliliter
MS	Mass Spectrometry
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NaI	Sodium Iodide
NJDEP	New Jersey Department of Environmental Protection
oz	ounces
PCB	Polychlorinated biphenyl
PDI	Pre-Design Investigation
POTW	Publicly Owned Treatment Works
PPE	Personal Protective Equipment
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPT	Radiation Protection Technician
RSO	Radiation Safety Officer

SD	Sediment
SOP	Standard Operating Procedure
SSHP	Site Safety and Health Plan
SVOC	semi-volatile organic compound
TRPH	Total Recoverable Petroleum Hydrocarbons
TSCA	Toxic Substances Control Act
UFML	USACE FUSRAP Maywood Laboratory
µR/hr	micro-Roentgen per hour (either abbreviation is acceptable)
USACE	U. S. Army Corps of Engineers
UST	Underground Storage Tank

1.0 SAMPLE TYPES

Field screening and sampling activities at the Formerly Utilized Sites Remedial Action Program (FUSRAP) Maywood Superfund Site (FMSS) may involve the sampling of soils, backfill materials, building materials, air, groundwater, sediments, surface waters, and wastewater. Task-specific Work Plans are developed for each task that involves sampling or analysis of samples. The task-specific Work Plan details the number and types of samples to be collected.

In addition to regular field samples, quality control (QC) and quality assurance (QA) samples will be collected. Field QC sample types include trip blanks, field blanks, temperature blanks, equipment rinsate blanks or wipe sample blanks, and duplicates. Matrix spike / matrix spike duplicates (MS/MSD) for organics and matrix spike / matrix duplicate (MS/MD) for inorganics are laboratory QC samples which will be collected in the field. The only field requirement for samples designated for MS/MSD (organic) analysis is that extra volume must be collected for aqueous samples (the quantity collected for soil or solid materials is typically more than adequate for meeting the required amount for MS/MSD). MS/MSD samples are further discussed in Sections 2.8, 4.4.2.1, and 6.2 of the Quality Assurance Project Plan (QAPP). QA samples will also be collected and analyzed by a qualified outside QA laboratory. QA samples are collected and analyzed to assess comparability.

Trip blanks accompany all volatile organic sample shipments. Volatile organic trip blanks consist of vials containing demonstrated organic-free water (water for which the concentration of organics is less than the method detection limit). Trip blanks are used for aqueous field samples. The aqueous vials are filled with zero headspace in the laboratory. The trip blank vials are shipped to the site, and then shipped back with the other volatile organic analysis (VOA) sample vials. The trip blank provides an indication of any contamination source arising from the vial itself or the trip back and forth to the lab. Trip blanks are only employed for samples to be analyzed for volatile organic compounds. There must be at least one trip blank in each shipping container that has volatile samples.

Field blanks are collected when dedicated equipment is used to determine whether the equipment is contributing contamination to the sample. A collection frequency of once per equipment lot is adequate for field blank samples. If a rinsate blank is already being collected, and the rinsate water comes into contact with the dedicated equipment, the field blank is not necessary.

Temperature blanks are water-filled bottles supplied by the Contract Laboratory for use in measuring the temperature of the samples upon arrival at the laboratory. For those samples required to be maintained at 4°C, a temperature blank must be included in each cooler shipped from or picked up at the FMSS.

Equipment rinsate blanks are samples of analyte-free water (water for which the concentration of all analytes is less than the method detection limit). The water is used to rinse sampling equipment after the equipment has been decontaminated, and then the water is added to the sample container for all analytes except radionuclides. For radionuclides, a wipe sample will be collected following decontamination of the sample collection equipment, and it will be analyzed using a suitable low background counter. Equipment rinsate blank test results provide information on the efficiency of the decontamination process. Equipment rinsate blanks must be collected each day that sampling occurs or each decontamination event, whichever is less, per matrix per parameter per field crew, per equipment type (assuming reusable sampling equipment is used). As a minimum, one rinsate blank will be collected daily per crew during groundwater sampling activities, unless disposable bailers are used. If disposable bailers are used, no rinsate blanks will be collected; however, a field blank shall be collected for each lot of bailers. Rinsate blanks for soil sampling will be collected every day that soil sampling is conducted. Where possible, the

rinsate blank should be collected following the decontamination of equipment used to sample at the location suspected to be the most highly contaminated. If deemed acceptable by USACE, equipment rinsates or wipe samples may be collected at a frequency of one per decontaminated lot of a given type of sample collection equipment.

Field duplicates are two samples collected from the same location (immediately adjacent to each other) at the same time in the same manner. Field duplicate test results provide a measure of overall sampling and test method precision as well as sample heterogeneity. Field duplicate samples must be identified or numbered so that the laboratory does not know the samples are duplicates (thus the term "blind duplicates"), thus eliminating potential bias in the test measurement. Field duplicate samples must represent no less than 10% of the total number of samples per matrix per parameter per area of study.

QA samples are split samples that are submitted to a qualified outside QA laboratory. Split samples are two portions of a sampled matrix that has been thoroughly homogenized. These samples are used to evaluate the performance of the Shaw Environmental, Inc. field crew and interlaboratory variability. QA and Contract Laboratory results will be compared by the Shaw or USACE Project Chemist. The results of the comparison will be discussed within the individual property Quality Control Summary Reports. QA samples shall be collected at approximately 5% of the total number of samples per matrix per parameter per area of study.

For the purpose of calculating the appropriate number of QC or QA samples, soil, sediment, groundwater, surface water, and building materials should be considered as separate matrices. Every attempt should be made to associate QC samples with field samples of similar matrix. This will only serve to improve the quality of the data obtained from QC sample test results.

2.0 ON-SITE OPERATIONS

Maywood site investigations and remedial actions will require use of a variety of sampling methods. The pre-design investigation, soil acquisition, remedial action tasks, groundwater remedial investigation, materials handling, and transportation / disposal will require soil, water, and / or building materials sampling. Procedures describing typical sampling methods, such as surface and shallow subsurface soil sampling, soil borings and sampling, concrete core sampling, building materials sampling and wipe sampling are provided in the EDMS (see Section 4.7 of the CDQMP QAPP). When individual tasks involve a type of sampling or other field data collection activity that is not addressed by the procedures in the EDMS, these additional procedures will be presented in the task-specific Work Plan.

Each task-specific Work Plan will provide a general description of the sample collection procedures to be followed as well as the task specific details that distinguish these procedures from the ones presented in this FSP. For each sampling method, the necessary support facilities will be identified. The discussion will focus on the procedures for addressing failures in the sampling system and the responsibilities for corrective action. A table, similar to **Tables 3-1** and **3-2** of this FSP, will be included which details the bottle requirements, preservation, and holding times to extraction and / or analysis for all analytical parameters and matrices.

The following sections discuss the proposed activities that are currently anticipated to require sampling.

2.1 RADIOLOGICAL FIELD SCREENING AND SOIL SAMPLING

2.1.1 Remedial Action Support Survey

The objective of the remedial action support survey is to detect the presence of residual activity at or below the derived concentration guideline level (DCGL) or cleanup level. Instruments shall be selected based on the detection capabilities for the contaminants and the DCGLs to be achieved. Remedial action support surveys are performed under the direction of the Site Radiation Safety Officer.

A walkover survey and subsurface measurements may be used to define the boundaries of the excavation. Initial walkover surveys will be performed using instruments capable of detecting elevated contamination levels under normal background conditions (5 to 9 micro-Roentgens per hour [µR/hr]). Examples include, but are not limited to the Bicron MicroREM meter, the Ludlum 12s, and the Ludlum 2221 mated to a Ludlum 44-10 Sodium Iodide (NaI) detector. The radionuclides at the FMSS, when present at elevated concentrations in surface and near surface soil, are capable of being detected with these instruments. For subsurface measurements, soil samples shall be collected using a direct push soil probe, test pitting, or hand auger technique. Downhole gamma logging will be accomplished, where feasible, using a NaI detector suspended down the soil probe, following collection of extracted soils. The depth of the soil probes will be dependent on existing site data and downhole gamma logging. Collected soils are packaged for transport to a designated location at the Maywood Interim Storage Site (MISS) for additional screening, sample collection, isotopic analysis and shipping / archiving, as required. Samples collected to meet formal Data Quality Objectives (DQOs) (e.g., Final Status Survey samples) are analyzed in a USACE/NJ Department of Environmental Protection (NJDEP) certified laboratory with 5% submitted to a QA lab for confirmatory analysis. Standard Operating Procedure (SOP) 509, "Soil Probe Investigation," located in the project EDMS, provides details of this activity.

Prior to excavation activities, land survey personnel will establish and stake the excavation boundaries based on the pre-design investigation data generated by Shaw.

During excavation, area gamma field scans will be performed and remedial support soil samples will be collected by Radiation Protection Technicians (RPTs). Remedial support surveying and sampling is performed under the direction of the Site Radiation Safety Officer (RSO). These data will be used to adjust the initial excavation boundaries and evaluate if an area is ready for Final Status Survey (FSS). Informal action levels for field instruments are established during the excavation by the RPT and the RSO. Action levels are considered guidance and based on area background conditions, "shine" from unexcavated contaminated soils, established DCGLs (Derived Concentration Guidance Levels), and process knowledge. Action levels are modified by the RPT with concurrence from the RSO as necessary, based on an evaluation of remedial support soil samples data from the USACE FUSRAP Maywood Laboratory (UFML) against established DCGLs and gamma field scan readings. RPTs typically use spray paint, pin flags, or verbal direction to Construction personnel to identify areas where additional excavation is required to satisfy clean-up goals. Gamma scans are used to determine bias locations for collection of remedial support soil samples. These samples are then collected and analyzed in the on-site lab to determine if an area is ready for final status survey. This process is referred to as "guiding the excavation."

> *SAFETY NOTE: RPTs will not guide excavations greater than 5 feet deep unless appropriate protective systems are in place. Alternate monitoring techniques using long-reach sampling and gamma scanning devices may be used to limit personnel entry into deep excavations. Personal safety shall not be compromised for the performance of radiological surveys.*

2.1.2 Final Status Surveys

Final Status Surveys are designed to demonstrate that the concentrations of radionuclides of concern in soil are compliant with the commercial use or residential use cleanup criteria, as appropriate. Final Status Surveys shall be performed according to the requirements established in the *Master Final Status Survey Plan* (USACE 2001a) and property-specific addenda. The Master FSS Plan is based on methodologies established in Environmental Protection Agency (EPA) document 402-R-97-016, the *Multi-Agency Radiation Survey and Site Investigation Manual* (MARSSIM), and the MARSSIM Revision 1, Corrections pages (EPA 2002).

2.2 MONITORING

Monitoring is necessary to evaluate possible changes in site conditions and track the potential risks to site workers and the adjacent population. An environmental monitoring program is conducted annually. The key elements of this program are:

- Measurement of external gamma radiation.
- Measurement of radon gas concentrations in air (from both radon-220 and radon-222).
- Sampling and analysis of streambed sediment for radioactive constituents and metals.
- Sampling and analysis of surface water for radioactive constituents and metals.
- Sampling and analysis of groundwater for radioactive constituents, metals, and volatile organic compounds.

In 2009, the sampling and analysis of sediments and surface waters for metals, and of groundwater for metals and volatile organics, will be discontinued. Radon monitoring is conducted at surface and subsurface soil locations that could create hazardous radon levels; e.g., within the structures over inaccessible soils. This radon monitoring is conducted in accordance with SOP 306, "Radon Sampling" (see project EDMS), or as specified in Radiation Protection Program implementing procedure PP-8-803, "Measurement of Airborne Radioactivity," located in the FMSS *Site Safety and Health Plan* (SSHP),

Appendix C (USACE 2006a). Additional monitoring, including meteorological monitoring and personal and area air particulate and organic vapor monitoring, is employed to track the potential risks to site workers and the adjacent population. Perimeter monitoring is further discussed in the General Environmental Protection Plan (GEPP) and the FMSS SSHP (USACE 1999b & 2006a, respectively). Monitoring data is used to calculate maximum doses from external gamma radiation and inhalation of radioactive particulates and radon gas. Guidance on monitoring use and monitoring methods for health and safety monitoring (work zone and breathing zone) are provided in the FMSS SSHP (USACE 2006a).

2.3 GROUNDWATER SAMPLING

Additional groundwater sampling was necessary to further characterize the FMSS. To date there have been two phases of groundwater sampling, called a groundwater remedial investigation (GWRI), conducted to determine the possible impact of site contaminants. The results of that investigation have shown principally the presence of a benzene plume and arsenic / lithium plumes in bedrock. Small plumes of limited dimensions have been identified in bedrock groundwater. Additional sampling to better characterize the source and extent of the benzene plume occurred during the summer of 2002 and winter of 2003.

Groundwater samples will be collected from existing and newly installed monitoring wells. Prior to sample collection, a synoptic round of groundwater level measurements will be obtained from selected wells. Samples of the well evacuation discharge water will be collected and analyzed for disposal purposes.

Groundwater samples will be collected using either of the following sampling equipment: submersible pump or Teflon® (or Teflon®-lined) bailer. In those instances where the volume of water present in the well is insufficient to collect a sample using the aforementioned sampling apparatus, a peristaltic pump will be used to collect the sample. The collected groundwater samples will be analyzed as specified in the RI Addendum – Proposed Source and Plume Delineation Work Plan. In accordance with the EPA Region II Low Stress (Low Flow) Sampling and Purging Procedure, March 1998, groundwater samples will be collected after field parameters stabilize. The parameters required for monitoring and applicable criteria are outlined in procedure 304 within the project EDMS, Purging and Sampling of Monitoring Wells.

2.4 WASTE SAMPLING

During the course of this project, various types of wastes may require sampling for characterization, including investigation-derived waste (IDW), soil or debris designated for offsite disposal, excavation groundwater, decontamination wastewater, stormwater, and dewatering effluent. Final disposition of these wastes will depend on the results of the sampling and analyses performed, relative to the permanent disposal facilities' waste acceptance criteria. Other wastes may also be encountered or generated such as building materials, rags, batteries, oily waste, Toxic Substances Control Act (TSCA) waste, and other special waste. The task-specific Work Plans will detail the types of wastes anticipated to be generated and how they will be handled. The following subsections discuss several common categories of waste anticipated for this project.

2.4.1 Investigation-Derived Waste (IDW)

During site investigations, IDW in the form of personal protective equipment (PPE), drill cuttings from well installations and purge and decontamination water are generated. Every effort will be made to minimize waste generated. All IDW will be containerized in approved containers. Soils, waters, and other materials will be containerized separately and the contents identified with weather-resistant labels affixed to each container's exterior. This IDW will be transported to the MISS, and sampled and tested as

necessary prior to disposal to determine a proper disposal method. Techniques for sampling IDW in drums can be found in the project EDMS, SOP 313, "Drum Sampling."

Laboratory analysis of the waste will be conducted as necessary to both characterize the waste and meet the requirements of the designated disposal facility. Wastes will be managed and transported for disposal in accordance with Shaw's Materials Handling, Transport, & Disposal (MHT&D) Plan and SOP 505, "Cuttings and Fluids Management" (USACE 1999a).

2.4.2 Soils and Debris Sampling Prior to Offsite Transport

All project solid, hazardous, and radiological waste will be disposed of in an offsite licensed / permitted disposal facility in accordance with local, state, and federal regulations. Radiologically contaminated soils and debris will be transported by rail to a permitted radiological disposal facility. Shaw will use any existing profile information and collected analytical data to properly manage and ship the waste soils from the Maywood Superfund Site.

Should additional characterization be required, materials will be sampled prior to transport to a selected disposal facility. The procedures for sampling these soils and debris will be, SOP 315, "Railcar Sampling" and SOP 311, "Building Materials Sampling," with minor modifications, both found within the project EDMS. The tests performed on the sampled materials will depend on the selected disposal facility's regulatory requirements for accepting the materials.

Soils being shipped to a USACE-approved facility must be tested according to the MHT&D Plan, Appendix A, which describes a typical Waste Acceptance and Management Plan (USACE 1999a).

2.4.3 Potentially Contaminated Wastewater

Potentially contaminated wastewater will be generated during project activities. Sources of potentially contaminated wastewater in project-controlled areas include, but are not limited to the following sources: equipment/personnel decontamination, storm water accumulation in contaminated areas, groundwater infiltration into contaminated areas, and from dust suppression activities. Potentially contaminated wastewater is typically pumped from the source (i.e., the point of accumulation) to on-site holding tanks, processed via an industrial treatment system, and discharged to a Publicly Owned Treatment Works (POTW) via the local sanitary sewer system. Discharge parameters and sampling frequencies are established in an Industrial Pre-Treatment Program permit, issued by the Bergen County POTW, and maintained in the project EDMS. Radionuclide discharge parameters are consistent with the maximum contaminant levels established in the EPA National Primary Drinking Water Regulations (49 CFR 166). Further discussion of potentially contaminated wastewater sources and management practices is located in the GEPP (USACE 1999b) and the Water Management Plan (USACE 2001c).

3.0 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

When handling samples in the field and in the laboratory, care should be taken to ensure the integrity of the samples is maintained at all times. Samples must be collected, transported, and received under strict chain of custody protocols consistent with procedures established by the EPA for litigation-related materials. Each task-specific Work Plan will provide a general description of the provisions for sample handling, as determined based on the nature of the samples and the maximum allowable holding times. The following subsections discuss general procedures for handling samples both in the field and in the laboratory.

3.1 FIELD PROCEDURES

The following subsections describe the steps to be taken in the field when collecting samples and preparing them for shipment to the analytical laboratory.

3.1.1 Sample Homogenization

Samples for all parameters except volatile organics will require homogenization after collection. For soil samples, the soil will be homogenized upon removal from the ground or waste pile. Water samples will be collected in such a way so as to be most representative of the source water. Homogenization ensures that all samples from a given area will have similar textures or characteristics, thus reducing potential biases from physical sample characteristics. The correct homogenization techniques, including sample handling and mixing and potential problems, are provided in the USACE Engineering Manual (EM) 200-1-3 Section E-2, (USACE 2001b). These techniques shall be used on the Maywood project.

3.1.2 Compositing Samples

Compositing techniques such as flow-proportioned and time composites (for sampling flowing surface waters, if required), and aerial and vertical composites, to be used for compositing soil samples for radiological and chemical analysis, are described in EM 200-1-3 Section E-3 (USACE 2001b). Compositing ensures that samples are representative. It also can reduce analytical costs. It is important that all composite samples be collected in the same way to avoid sampling bias. Compositing shall only be considered for off-site disposal purposes. Air sample filters may be composited according to methodologies established in the FMSS SSHP (USACE 2006a).

3.1.3 Field Preservation

Depending on the analyses proposed for the samples collected, chemical field preservation may be required. This preservation will be conducted in accordance with the requirements of SW-846 (EPA 1997). The preservative will be noted both on the sample labels and on the respective chain-of-custody (COC) for the sample. When field preservation is required for samples collected during a particular task, this effort will be further detailed in the task-specific Work Plan. Sample preservation techniques for all sampling and test methods that may be employed on this project are provided in **Tables 3-1** and **3-2**.

As a minimum, all samples being analyzed for chemical analyses will be placed on ice immediately following collection. The temperature and trip blanks as required will be placed in the coolers at the same time as the ice (i.e., at the start of the sampling event) to ensure they receive adequate cooling and are

representative of the temperature of the samples prior to transferring custody of the samples to the analytical laboratory.

3.1.4 Equipment Decontamination

Maywood field personnel will follow the requirements in SOP 506, "Decontamination" (located in the project EDMS) and EM 200-1-3 Section E-5 (USACE 2001b) for decontamination of sampling equipment. The goal of decontamination is to eliminate cross-contamination resulting from the use and reuse of sampling equipment. Decontamination waste will be managed in accordance with SOP 505, "Cuttings and Fluids Management" and the Materials Handling, Transport and Disposal (MHT&D) Plan (USACE 1999a).

3.1.5 Packaging and Shipping

Each sample bottle will be identified with a separate identification label. Labels may be pre-printed and / or augmented by notations made in indelible / waterproof ink. Entry errors will be crossed out with a single line, dated, and initialed. Each securely fixed label will include the following:

- Project identification
- Sample identification
- Preservatives added
- Type(s) of analysis(es) to be performed
- Date and time of collection

Table 3-3 details the sample numbering format to be used for the project. Each sampling task or area will have a separate designation.

A properly completed COC form shall accompany all sample shipments. The COC description section requires the following:

- Sample identification number
- Container types and preservation techniques
- Date and time of collection
- Analytical test parameters or test parameter method (in Analysis / Remarks Section); e.g., EPA Method 8270. Also indicate lab QC sample, if applicable; e.g., MS/MSD, rinseate blank or RB, etc. This is very important so that the laboratory does not accidentally select the wrong sample to represent a QA sample
- Specific instructions to the lab; e.g., unique turnaround times, specific analytes or other special instructions for analysis
- Number of containers corresponding to each sample ID number and parameter
- Specific sample collection method (grab or composite)
- Type of matrix
- Names of the sampler(s) and signature of the person shipping the samples
- Date and time that the samples were sealed for delivery

- Signature of the individual receiving the samples at the laboratory (to be filled out at the laboratory)

The original record will accompany the shipment, and copies will be retained by the sampler for return to project management and the project file. Whenever split samples are collected for comparison analysis by a qualified QA Laboratory, a separate COC is prepared for those samples and marked to indicate that they are USACE QA samples. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record will document transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to / from a secure storage area. An example of the COC form to be used for these investigations is provided in Appendix C of the QAPP.

Environmental samples will be transported to the Contract Laboratory and QA Laboratory via the most rapid means. If the Contract Laboratory is located within approximately 100 miles of the FMSS, they (the Laboratory) will pick up samples by courier service. Otherwise overnight delivery service will be utilized. Each task-specific Work Plan will detail the transport means to be used for the samples. All shipments of samples will be in compliance with applicable United States Department of Transportation (DOT) regulations.

Shipment of samples on Friday to the Contract Laboratory will be discouraged unless it is absolutely necessary and the laboratory has confirmed that personnel will be present on Saturday to receive and implement any necessary processing within the analytical holding times. No samples shall be shipped for weekend or holiday delivery at the QA Laboratory. Therefore, samples designated for the QA Laboratory that are collected on Friday or during the weekend must be properly preserved and held in the field until they can be shipped to arrive on the next business day (assuming that holding times will not be adversely impacted).

EM 200-1-3 Section F-2 (USACE 2001b) and SOP 504, "Labeling, Packaging, and Shipping Environmental Samples," (located in EDMS) detail further the requirements for packaging and shipping samples.

Radiological samples will normally be shipped as excepted packages for limited quantities of radioactive materials as defined in 49CFR173.421. Guidelines for shipment of these samples are contained in SOP 508, "Procedure for Shipping Radiologically Contaminated Environmental Samples" (located in EDMS). Chemical samples will be shipped as excepted packages for limited quantities of miscellaneous hazardous materials as defined by 49CFR173.155. Guidelines for shipment of these samples are contained in SOP 504, "Labeling, Packaging, and Shipping Environmental Samples," located in EDMS.

3.2 LABORATORY SAMPLE HANDLING

The following subsections describe the steps the laboratory must take as part of the process of preparing samples for analysis.

3.2.1 Laboratory Subsamples

The Contract laboratories shall use proper techniques as presented in Section E-4 of EM 200-1-3 (USACE 2001b) to sample an aliquot or portion of the field sample received into the laboratory. Generally, except for volatile organic samples, field samples should be thoroughly mixed (shaken in the case of water samples) prior to subsampling. Dealing with potential problems caused by multi-layer liquid samples is also discussed.

3.2.2 Laboratory Custody Procedures

Laboratory custody procedures, along with the holding times and sample preservative requirements for samples, will be described in laboratory QA Plans (see the project EDMS for examples). These documents will identify the laboratory custody procedures for sample receipt and log-in, sample storage, tracking during sample preparation and analysis, and laboratory storage of data.

3.2.3 Cooler Receipt

The condition of shipping coolers and enclosed sample containers, including the results of all checks for temperature and pH preservation, will be documented upon receipt at the analytical laboratory. This documentation will be accomplished using the Cooler Receipt Checklist presented in Appendix C of the QAPP, or an equivalent checklist. A copy of the checklist will be faxed to the Project Chemist immediately after it has been completed at the laboratory. A copy of the completed checklist will also be transmitted with the final analytical results from the laboratory.

Table 3-1
Typical Soil Sample Containers and Associated Requirements for Maywood

Analyte Group	Container	Minimum Sample Size	Preservative	Holding Time[a]
Volatile Organic Compounds	4 – EnCore Samplers	5 g per EnCore	Cool, 4°C	48 hrs (extraction) 14 d (analysis)
Semivolatile Organic Compounds	1 - 8 oz glass jar with Teflon®-lined cap	150 g	Cool, 4°C	14 d (extraction) 40 d (analysis)
Pesticides / Polychlorinated Biphenyls (PCBs)	Use same container as semi-volatile organic chemicals (SVOCs)	150 g	Cool, 4°C	14 d (extraction) 40 d (analysis)
Herbicides	1 - 4 oz glass jar with Teflon®-lined cap	50 g	Cool, 4°C	
Metals, Cyanide, and Rare Earth Elements	1 - 4 oz wide mouth plastic or glass jar	20 g	Cool, 4°C	180 d, Hg at 28 d
Total Recoverable Petroleum Hydrocarbons (TRPH)	2-1 Liter Amber Boston Round	1000 mL	Cool, 4°C HCl	Analyze ASAP
Waste Characteristics	1 - 16 oz wide mouth glass jar with Teflon®-lined cap	1000 g	Cool, 4°C	general 14 d
Radiochemical Parameters	1 - 32 oz wide mouth plastic jar with unlined plastic cap	560 g	None	180 d
Grain Size	1 - 8 oz glass jar	350 g	None	None

Notes:
a : Holding times for extractions, and for analyses (for methods without an extraction holding time requirement) are calculated from the time of sample collection. Holding times for analyses, for methods involving an extraction step, are calculated from the time of extraction to the time of analysis

Table 3-2
Container Requirements for Water Samples for the Maywood Investigations

Analyte Group	Container	Minimum Sample Size	Preservative	Holding Time
Volatile Organic Compounds	2 - 40 mL glass vials with Teflon®-lined septum (no headspace)	40 mL	HCL to pH <2 Cool, 4°C	14 d
Semivolatile Organic Compounds	2 - 1L amber glass bottle with Teflon®-lined lid[1]	1000 mL	Cool, 4°C	7 d (extraction) 40 d (analysis)
Pesticides / PCBs and Herbicides	3 - 1L amber glass bottle with Teflon®-lined lid[1]	1000 mL	Cool, 4°C	7 d (extraction) 40 d (analysis)
Metals and Rare Earth Elements	1 - L poly bottle	500 mL, metals 200 mL, Hg	HNO_3 to pH <2	180 d, metals 28 d, Hg
Dissolved Organic Carbon	125 mL poly bottle	50 mL	H_2SO_4 to pH <2 Cool, 4°C	28 d
pH	125 mL poly bottle	50 mL	Cool 4°C	
Cyanide	1 - L polybottle	500 mL	NaOH to pH >10 Cool, 4°C	14 d
TRPH	1 - L glass bottle	1000 mL	H_2SO_4 to pH <2 Cool, 4°C	28 d
Alkalinity/TDS/TSS	1 - L polybottle	100 mL ea.	Cool, 4°C	7 d
Total Recoverable Phenolics	1 - L glass bottle	500 mL	H_2SO_4 to pH <2 Cool, 4°C	28 d
Oil & Grease	1 - L glass bottle	1000 mL	H_2SO_4 to pH <2 Cool, 4°C	28 d
MBA Surfactants	500 mL poly bottle	250 mL	Cool, 4°C	48 h
BOD 5-Day	1 - L glass bottle	1000 mL	Cool, 4°C	48 h
COD	125 mL poly bottle	50 mL	H_2SO_4 to pH <2 Cool, 4°C	28 d
Sulfate	125 mL poly bottle	50 mL	Cool, 4°C	28 d
Dissolved Sulfide	500 mL poly bottle	500 mL	Cool, 4°C add 2 mL zinc acetate plus NaOH to pH > 9	7 d
Ammonia Nitrogen	500 mL poly bottle	400 mL	Cool, 4°C H_2SO_4 to pH <2	28 d
Nitrate	125 mL poly bottle	100 mL	Cool, 4°C	48 h
Ortho Phosphorus	125 mL poly bottle	50 mL	Cool, 4°C H_2SO_4 to pH <2	28 d
Methane	40 mL glass vial	5 mL	Cool, 4°C	14d
Ignitability (flash point)	500 mL boston round	100 mL	None	Analyze ASAP
Reactivity	500 amber boston round	250 mL	Cool, 4°C	Analyze ASAP
Radiochemical Parameters	2 - 1 gal plastic containers[1]	4 L	HNO_3 to pH <2	180 d

Notes:
1 One investigative water sample in twenty will require an additional volume for the laboratory to perform appropriate laboratory QC analysis. (i.e., MS/MSD).

Table 3-3
Sample ID System for All Maywood Sites

Site Designator		Activity Designator		Field Measurement / Sample Collection Designator		Station Number	Media		Sample Type		Sequential Sample Number	
X X X[1,2]		AAA[1]		V V[1]		N N N	mm[1]		n[1]		# # # # # #[1,2]	
000	= Laboratories	BKG	= Background	DG	= Down Hole Gamma	Unique Station No.	BZ	= Air Filter (Breathing Zone)	0	= Regular	000001 to 002000	Test Pits
001	= Maywood Site	XBP	= Burial Pit	DP	= Direct-push		AE	= Air Filter (Environmental)	1	= Duplicate	002001 to 019949	PDI
01a	= 72 Sidney Street	XDR	= Drum	GB	= Surface Grab		GA	= Air Filter (General Area)	2	= Spit	019950 to 019999	Asbestos Pile Removal
02a	= 100 Hancock Street	EMP	= Environmental Monitoring Plan	GP	= Soil Probe		AB	= Aquatic Biota	3	= Trip Blank	020000 to 020499	Swale
02b	= 80 Hancock Street	EPP	= Environmental Protection Plan	MW	= Monitoring Well		BM	= Building Materials	4	= Equipment Rinsate	020500 to 021999	GWRI
02c	= 80 Industrial Road	FSS	= Final Status Survey	PP	= 4 in. Push-pipe		GW	= Groundwater	5	= Site Source Water Blank		
02d	= 8 Mill Street	GWR	= Groundwater Remedial Investigation	SB	= Soil Boring		ID	= Investigation Derived Waste			022000 to 022077	General Environmental, SWEC
03a	= 170 Gregg Street	PDI	= Pre-design Investigation	SI	= Surface ISOCS®		RD	= Radon Detector			024500 to 029999	
04a	= 160/174 Essex Street (Bank of NY)	RAR	= Disposal Soil Characterization				SD	= Sediment				
04b	= I-80 Westbound ROW	XPT	= Pilot Test				SM	= Smears (wipes)			022078 to 024499	General Environmental, SEC
04c	= 150 Essex Street, L001	XRT	= Routine									
04d	= I-80 Eastbound ROW	SWL	= Swale									
05a	= 99 Essex Street (Muscarelle)	XTL	= Test Pit				ST	= Storm Water			030000 to 034999	Routine/Health & Safety
05b	= 113 Essex Street (Bank of NY)	WTD	Water Treatment and Disposal				SB	= Subsurface Soil			(Not Assigned)	Surface ISOCS®
05c	= 200 NJ Route 17 South						SS	= Surface Soil			035000 to 039999	Pilot Demonstration
06a	= 85-101 NJ Route 17 North						SW	= Surface Water			040000 to 049999	Soil Remediation
06b	= 137 NJ Route 17 North						TB	= Terrestrial Biota			(Not Assigned)	Drums
06c	= 167 NJ Route 17 North						TD	= TLDs			(Not Assigned)	GWRA
06d	= 239 NJ Route 17 North										050000 to 074999	Final Status Survey
06e	= **29 Essex St**.										075000 to 079999	Disposal
07a	= 111 Essex Street										080000 to 089999	Water Treatment
07b	= Hackensack & Lodi Railroad											
08a	= 23 West Howcroft Road											
09a	= 149-151 Maywood Avenue											
10a	= 100 West Hunter (Stepan)											
11a	= 205 Maywood Avenue											
11b	= 61 West Hunter Avenue											
11c	= 50 West Hunter Avenue											
12a	= NY, Susquehanna & Western Railway											
12b	= 100 West Hunter Avenue (MISS)											
12c	= NJ Route 17 ROW											
13a	= Transect H & I											
14a	= Transect B											
15a	= Transect C											
16a	= Transect D											
17a	= Transect G											
18a	= Transect J											
19a	= Parkway											
20a	= Grove											
21a	= Ballod											
22a	= Lodi Brook											
22b	= Lodi Brook Drainage Basin											
23a	= Westerly Brook											
23b	= Westerly Brook Drainage Basin											
24a	= Saddle River											
25a	= Coles Brook											
26a	= Saddle River County Park											
27a	= BCUA											
Other designators may be added as necessary.		Other designators may be added as necessary.		Other designators may be added as necessary.		Sequential number for based on each site (property).	Other designators may be added as necessary.				Sequential number based on each site (property), and type of activity.	

Notes: 1. XXX, AAA, VV, mm, n, ###### = Sample ID legend to be used for database reporting
2. XXX, ##### = Sample ID to be used for sample collection and delivery to lab.

This page intentionally left blank.

4.0 FIELD DOCUMENTATION

The following is a summary of the requirements for recording and documenting the conditions of the field work for the project. Emphasis is placed on sample designation and documentation procedures that will be followed during sample collection to ensure that samples collected can be easily tracked.

4.1 SAMPLE INFORMATION DOCUMENTATION

All information relating to collection of field samples, including personnel names, sampling method, location, conditions, date and time, sample numbers, and observations shall be recorded in a bound field notebook. COCs will be filled out in accordance with the FSP Section 3.1.5 and SOP 504, "Labeling, Packaging, and Shipping Environmental Samples" (located in the project EDMS). Daily Quality Control Reports (DQCRs) will also be used to document field activities on a daily basis. An example COC and DQCR can be found in the QAPP, Appendix C. Field boring logs and well diagrams, when required for a particular task, are also considered part of the field documentation. Examples of such documentation shall be included in task-specific Work Plans, if it is required for the task activity. Radiological survey maps and geographical information system (GIS) plots generated by the Radiation Protection Group may also be used to document sample locations or activities. An example of this is the final status survey gamma walkover map, which typically identifies the location of bias samples collected. The offsite subcontractor laboratory(ies) will have an information management system that will track the progress of the sample through the laboratory. The procedure for documentation of laboratory activities is in Section 6.0 of the offsite subcontractor laboratory's Quality Assurance Program Plan (see the project EDMS). The analogous documentation procedure for UFML is located within Section 7.0 of their Quality Manual (USACE 2006b).

4.2 PREPARATION OF FIELD LOGBOOKS

Shaw field logbooks are bound with lined, consecutively numbered pages. Separate field books, as necessary, will be maintained by each field team responsible for sampling and support activities. All pages must be numbered prior to initial use of the logbook. The following information shall be recorded inside the front cover of the logbook:

- Person and organization to whom the book is assigned (with signature), and phone number(s)
- Start Date
- Project Name
- Shaw Job Number
- Field Operations Manager's Name
- Sequential Book Number (if applicable)

All entries to a logbook will be made by the assigned person. Any transfer of the logbook to a different person will require a signature and date from both the original assignee and the new person. Field notes will include identification of field control samples such as duplicates, split samples, MS/MSDs, equipment rinsate blanks, etc. Monitoring instrument readings will also be noted in the logbook. Other specifics regarding logbook entries, corrections, and opinions are discussed in SOP 507, "Field Notebook Content and Control." The same requirements will be imposed on all Shaw field subcontractors. Copies of field logs will be kept with their respective analytical data reports and submitted with the reports to the USACE.

4.3 PHOTOGRAPHS

Photographs may be taken of sampled areas in support of the information written into the logbook. Photographs may be collected on film or digital media. Information typically included with photographic records includes: the date, time, weather conditions (if applicable), subject, purpose of taking photograph, person taking the photograph, and photograph identification number. Photographs will become part of the project files and are therefore subject to the documentation requirements of this CDQMP and the Contractor Quality Control Plan (USACE 2005) for the FMSS.

5.0 SAMPLING EQUIPMENT AND PROCEDURES

The sampling and field measurement procedures that may be used on FMSS investigation and remediation tasks are provided in the project EDMS. A list of the procedures is provided as Appendix A. The 300-series procedures describe a variety of soil and water sampling methods. These procedures describe sample types, equipment, and techniques. The 400-series of procedures address different field measurement techniques. The 500 series procedures in Appendix A describe sample custody, decontamination, sample packaging and shipping, and proper use and control of a field notebook. The 600 series pertain to sample receipt, preparation, testing, and overall QC for the Maywood On-site Laboratory. Procedures governing the operation of field radiological monitoring instruments and the performance of radiological surveys are established in the Radiation Protection Program, FMSS SSHP, Appendix C (USACE 2006a). Shaw recognizes that these procedures are subject to the approval of the USACE Project Manager for the FMSS.

Specific details of the application of these techniques to the individual tasks or other procedures that may be required as part of the tasks will be included in the task-specific Work Plans. Any changes to the procedures referenced herein will also be approved by the USACE prior to implementation. If changes are made, the revised procedure will be noted with a revision number and incorporated into the CDQMP along with the approval documentation received from the USACE.

This page intentionally left blank.

6.0 REFERENCES

1. EPA 1997, Environmental Protection Agency. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*, Version 2, December 1997.

2. EPA 1998, Environmental Protection Agency. *Region 02 Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells*, SOP#GW0001. March 16, 1998.

3. EPA 2002, Environmental Protection Agency. *Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)*. EPA 402-R-97-016. August 2002.

4. Stone & Webster 1998, Stone & Webster Environmental Technology & Services, Inc. *Maywood Environmental Remediation Contract Proposal*, Volume 4A: Site Project – Technical Portion. November 2, 1998.

5. USACE 1994, U.S. Army Corps of Engineers. *Requirements for the Preparation of Sampling and Analysis Plans*, EM 200-1-3. September 1994.

6. USACE 1999a, U.S. Army Corps of Engineers. *Materials, Handling, Transportation, and Disposal Plan*. Revision 01. Prepared for the USACE by Stone & Webster, Inc. October 1999.

7. USACE 1999b, U.S. Army Corps of Engineers. *General Environmental Protection Plan*. Prepared for the USACE by Stone & Webster, Inc. November 1999.

8. USACE 2001a, U.S. Army Corps of Engineers. *Master Final Status Survey Plan.* Prepared for the USACE by Stone & Webster, Inc. November 2001.

9. USACE 2001b, U.S. Army Corps of Engineers. *Engineering and Design - Requirements for the Preparation of Sampling and Analysis Plans*, EM-200-1-3, February 2001.

10. USACE 2001c. U.S. Army Corp of Engineers. *Water Management Plan*. Revision 2. Prepared for the USACE by Stone & Webster, Inc. November 2001.

11. USACE 2005, U.S. Army Corps of Engineers. *Contractor Quality Control Plan, Revision 1*. Prepared for the USACE by Shaw Environmental, Inc. August 2005.

12. USACE 2006a, U.S. Army Corps of Engineers. *Site Safety and Health Plan*, Revision 03. Prepared for the USACE by Shaw Environmental, Inc. June 2006.

13. USACE 2006b, U.S. Army Corps of Engineers. *UFML Quality Manual*, prepared for USACE by Shaw Environmental, Inc., April 2006.

14. DOE 1995, U.S. Department of Energy. *Engineering Evaluation / Cost Analysis (EE/CA) for the Cleanup of Residential and Municipal Vicinity Properties at the Maywood Site, Bergen County, New Jersey*. September 1995.

This page intentionally left blank.

APPENDIX A
FIELD PROCEDURES LIST

This page intentionally left blank.

MAYWOOD PROCEDURES LIST

The following is a list of procedures that will be used in conducting field sampling, testing, and associated activities at the Maywood site.

100 SERIES – SURVEYING AND SCREENING

SW-MWD-101-1	SURVEYING
SW-MWD-102-1	DOWNHOLE GAMMA RADIATION LOGGING
SW-MWD-103-0	ROUTINE OPERATIONS PROCEDURE FOR ISOCS MEASUREMENTS OF SURFACE SOIL - **DELETED**
SW-MWD-104-0	METHOD QC PROTOCOL FOR FIELD MEASUREMENTS USING CANBERRA'S IN-SITU GAMMA SPECTROMETER **-DELETED**
SW-MWD-105-1	SOIL GAS SURVEYS
SW-MWD-106-0	ISOCS PROCEDURE FOR IMPLEMENTATION OF QC PROTOCOL - **DELETED**
SW-MWD-107-0	EXPOSURE RATE INSTRUMENTS - *MOVED TO RADIATION PROTECTION PLAN*
SW-MWD-108-1	GLOBAL POSITIONING SYSTEM (GPS) SURVEY
SW-MWD-110-0	DATA VALIDATION PROTOCOL FOR FIELD DATA COLLECTED USING CANBERRA'S IN-SITU GAMMA SPECTROMETER - **DELETED**
SW-MWD-112-0	CONTROL OF SEALED RADIOACTIVE SOURCES - *MOVED TO RADIATION PROTECTION PLAN*

200 SERIES – GEOTECHNICAL

SW-MWD-201-1	AQUIFER TESTING
SW-MWD-202-0	*RESERVED*
SW-MWD-203-1	ROCK CORING
SW-MWD-204-1	SLUG TESTING
SW-MWD-205-1	PACKER TESTING

300 SERIES – SAMPLING

SW-MWD-301-1	SEDIMENT SAMPLING
SW-MWD-302-1	SURFACE WATER SAMPLING
SW-MWD-303-1	MONITORING WELL INSTALLATION AND DEVELOPMENT
SW-MWD-304-1	PURGING AND SAMPLING MONITORING WELLS
SW-MWD-305-0	RESERVED
SW-MWD-306-1	RADON SAMPLING

SOIL AND BUILDING MATERIALS SAMPLING

SW-MWD-307-1	SURFACE AND SHALLOW SUBSURFACE SOIL SAMPLING
SW-MWD-308-1	SOIL BORINGS AND SAMPLING
SW-MWD-309-1	CONCRETE CORE SAMPLING
SW-MWD-310-0	RESERVED
SW-MWD-311-1	BUILDING MATERIALS SAMPLING
SW-MWD-312-1	WIPE SAMPLING PROCEDURES
SW-MWD-313-1	DRUM HANDLING AND SAMPLING

400 SERIES - FIELD MEASUREMENT PROCEDURES

SW-MWD-401-1	OPERATION OF THE MULTIRAE MULTI-GAS MONITOR

SW-MWD-402-1 OPERATION OF THE TVA-1000 PHOTOIONIZATION/FLAME IONIZATION DETECTOR
SW-MWD-403-1 OPERATION OF THE TSI DUST TRAK™ AEROSOL MONITOR
SW-MWD-404-1 RESERVED
SW-MWD-405-1 CALIBRATION AND MAINTENANCE OF TURBIDIMETER
SW-MWD-406-1 CALIBRATION AND MAINTENANCE OF ORION PLATINUM EH ELECTRODE
SW-MWD-407-1 CALIBRATION AND MAINTENANCE OF IN-SITU HERMIT ENVIRONMENTAL DATA LOGGER
SW-MWD-408-1 CALIBRATION AND MAINTENANCE OF SOLINST INTERFACE METER
SW-MWD-409-1 ON-SITE WATER QUALITY TESTING
SW-MWD-410-1 GROUNDWATER LEVEL MEASUREMENT

500 SERIES – MISCELLANEOUS

SW-MWD-501-1 CONDUCTING FIELD AUDITS
SW-MWD-502-0 *RESERVED*
SW-MWD-503-1 OVERBURDEN DRILLING METHODS
SW-MWD-504-1 LABELING, PACKAGING, AND SHIPPING ENVIRONMENTAL SAMPLES
SW-MWD-505-1 CUTTINGS AND FLUIDS MANAGEMENT
SW-MWD-506-1 DECONTAMINATION
SW-MWD-507-1 FIELD NOTEBOOK CONTENT AND CONTROL
SW-MWD-508-1 PROCEDURE FOR SHIPPING RADIOLOGICALLY CONTAMINATED ENVIRONMENTAL SAMPLES
SW-MWD-509-1 SOIL PROBE INVESTIGATION

600 SERIES – ONSITE LABORATORY

SW-MWD-601-2 LABORATORY INTERNAL DATA EVALUATION: QUALITY ASSURANCE AND CORRECTIVE ACTION
SW-MWD-602-1 *RESERVED*
SW-MWD-603-2 RADIOCHEMISTRY LABORATORY DOCUMENT DEVELOPMENT AND CONTROL
SW-MWD-604-2 SAMPLE IDENTIFICATION AND STORAGE
SW-MWD-605-2 SAMPLE CONTAINER CONTROL
SW-MWD-606-3 REAGENT, SOLUTION, AND STANDARD PREPARATION
SW-MWD-607 through 610 - *RESERVED*
SW-MWD-611-2 LABORATORY CONTAMINATION CONTROL
SW-MWD-612-2 SAMPLE DISPOSAL
SW-MWD-613-3 HAZARDOUS WASTE MANAGEMENT
SW-MWD-614 THROUGH 621 - *RESERVED*
SW-MWD-622-4 ORTEC GAMMA SPECTRUM ANALYZER OPERATION
SW-MWD-623-3 OPERATION OF THE PROTEAN WPC-9550 ALPHA-BETA GAS PROPORTIONAL AUTOMATIC PLANCHET COUNTER
SW-MWD-624-2 ORTEC ALPHA SPECTROSCOPY SYSTEM OPERATION
SW-MWD-625-4 PROTEAN GAS PROPORTIONAL COUNTER MULTI-DETECTOR SYSTEM OPERATION
SW-MWD-626 THROUGH 634 - *RESERVED*
SW-MWD-635-3 METHODOLOGY AND GUIDELIINES FOR ANALYTICAL AND TOP LOADING BALANCE OPERATIONS
SW-MWD-636 THROUGH 640 - *RESERVED*
SW-MWD-641-1 RECEIPT AND PREPARATION OF SOIL SAMPLES FOR ANALYSIS BY GAMMA SPECTROMETRY

SW-MWD-642 - *RESERVED*
SW-MWD-643 -1 RECEIPT AND PREPARTION OF WATER SAMPLES FOR ANALYSIS
SW-MWD-644 THROUGH 645 - *RESERVED*
SW-MWD-646-3 URANIUM AND THORIUM ANALYSIS SEPARATION METHOD
SW-MWD-647-3 URANIUM ANALYSIS SEPARATION METHOD
SW-MWD-648-3 THORIUM ANALYSIS SEPARATION METHOD
SW-MWD-649-3 RADIUM 226 ANALYSIS SEPARATION METHOD
SW-MWD-650-4 RADIUM 228 SEPARATION FOR ISOTOPIC ANALYSIS – EPA 904
SW-MWD-650A - *RESERVED*
SW-MWD-651-2 PREPARATION OF SAMPLES FOR GROSS ALPHA AND GROSS BETA MEASUREMENT
SW-MWD-653-2 COPRECIPITATION METHOD FOR GROSS ALPHA RADIOACTIVITY IN DRINKING WATER
SW-MWD-654-1 KINETIC PHOSPHORESENCE ANALYZER (KPA) METHOD FOR MEASUREMENT OF URANIUM IN DRINKING WATER
SW-MWD-655 THROUGH 659 - *RESERVED*
SW-MWD-660-1 URANIUM ANALYSIS SEPARATION METHOD FOR WASTWATER
SW-MWD-661-2 THORIUM ANALYSIS SEPARATION METHOD FOR WASTWATER
SW-MWD-662-3 RADIUM-226 ANALYSIS SEPARATION METHOD FOR WASTEWATER
SW-MWD-663-1 RADIUM-228 SEPARATION FOR ISOTOPIC ANALYSIS METHOD OF WASTEWATER SAMPLES
SW-MWD-664-1 PREPARATION OF WASTEWATER SAMPLES FOR GROSS ALPHA AND GROSS BETA MEASUREMENT

This page intentionally left blank.

APPENDIX B
SECTION E AND F OF EM 200-1-3, REQUIREMENTS FOR THE PREPARATION OF SAMPLING AND ANALYSIS PLANS

This page intentionally left blank.

Appendix E
Sample Manipulation Instructions

E.1 Filtration Techniques (Liquid Media)

E.1.1 Scope and application. This instruction outlines two different techniques for the filtration of aqueous media (i.e., ground water, surface water, and potable water). The procedures address in-line filtration, where the filter assembly is under positive pressure, and vacuum filtration, where the filter assembly is under negative pressure. In addition, the procedures describe and recommend specific filtration equipment. Filtration of aqueous samples is performed when the removal of sill algae, particulates, and other debris is desired. Predominantly, filtration is employed when water samples are to be tested for dissolved metals. However, many regulatory agencies no longer accept filtered samples as representative of "dissolved metals" concentrations. Therefore, the position of the regulators should be investigated to assure acceptance of the data generated from these types of samples. An alternative procedure to filtration may be the use of low-flow sampling techniques. Filtered samples for metals (dissolved fractions) should be analyzed in conjunction with nonfiltered samples to determine the metal concentration in solution versus metals associated with solids. Analysis of both filtered and unfiltered samples will allow the determination of metal concentration associated with the solid. Samples requiring organic analyses are not filtered unless specifically requested in the field sampling plan. Filtration techniques for ground water should be conducted in the field shortly after collection and before the addition of preservatives. A delay in the filtration of these samples may allow potential changes in carbon dioxide and oxygen concentrations, effectively changing the water pH and Eh, and leading to metals precipitation. These particulates are then erroneously filtered and may lead to a negative bias in the filtered sample results.

E.1.2 Filtration techniques. The following instructions will focus on in-line positive and negative pressure filtration of aqueous media. In-line filtration is recommended because it provides better consistency through less sample handling and minimizes sample exposure to the atmosphere. In the instructions, specific types of filtration devices will be referenced. For assessment of dissolved concentrations of major ions and trace metals, 0.1- m filters are recommended, although 0.45- m filters are normally used for most regulatory programs. In addition, analytical methods used to determine dissolved metal concentrations have historically used 0.45- m filters to separate dissolved and particulate phases. Therefore, if the filter pore size is changed, comparability between existing and newly generated data must be evaluated. Filters must be prerinsed following manufacturers' instructions. When no recommendations for prerinsing exist, pass a minimum of 1 L of water through the filter prior to sampling. For ground water this is done after purging is complete and before the sample collection.

E.1.2.1 Positive pressure filtration. Positive pressure filtration methods are preferred for aqueous sample filtration. Aqueous samples that may require positive pressure filtration include ground water samples, surface water samples, and potable water supply samples. To filter an aqueous sample using the positive pressure technique, a pump, filter, and tubing are required. The following are examples of equipment that may be used for positive pressure in-line filtration.

E.1.2.1.1 Pump system.

- High-flow range: 3 - 2,300 mL/min
- Low-flow range: 6 - 460 mL/min
- System flow control: ±10%

E.1.2.1.2 Filter assembly.

- Groundwater sampling capsule: 6 to 12 mm (1/4 to 1/2 in.), tapered barb fitting
- Pore size: 0.45 mm or as dictated by project
- Continuous use pressure: 413.6 kPa (60 psi) at ambient conditions
- Maximum momentary pressure: 689 kPa (100 psi) at ambient conditions

E.1.2.1.3 Filtration procedure.

Use polytetrafluoroethylene (PTFE) (PTFE is commonly referred to using the registered name of Teflon) tubing for pump and filter connections.

Connect the appropriately sized in-line filter to the discharge tubing from the pump. Make sure the flow arrow on the filter is pointing in the correct direction and the system is leakproof.

Apply pressure to the liquid sample (via pump) to force it through the filter directly into the appropriate sample container at a pressure recommended by the equipment manufacturer.

Replace the in-line filter when the flow becomes too restricted because of a buildup on the filter. To replace the filter, discontinue pumping (turn off pump), relieve the pressure in the system (line between the pump and the filter), and disconnect the filter and replace with a new one.

- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.

  Verify that a PTFE liner is present in the cap. Secure the cap tightly.

- Label the sample bottle with an appropriate label. Be sure to include all necessary information.
- Record the information in the field logbook and field sheet, and complete all chain-of-custody documents (see Instruction F-1, "Documentation," Appendix F).
- Release the pressure in the filtration equipment, disconnect sample filtration device from the discharge tubing, and thoroughly decontaminate or properly dispose of all equipment and materials in accordance with the project sampling and analysis plan.

E.1.2.2 Negative pressure filtration. Aqueous samples that may require negative pressure filtration include ground water samples, surface water samples, and potable water supply samples. To filter an aqueous sample using the negative pressure technique, a pump, filter, sample collection container, and tubing are required. The following equipment may be used for negative pressure (vacuum) filtration:

E.1.2.2.1 Pump system, hand-operated vacuum/pressure pump

- Maximum vacuum: 25 in. Hg
- Maximum pressure: 103.4 kPa (15 psi)
- Composition: metal or polyvinyl chloride (PVC)

E.1.2.2.2 Filter assembly, Nalgene filter funnel/collection flask

- Filter composition: cellulose nitrate
- Pore size: 0.45 m or as dictated by project
- Collection flask capacity: 500 mL (16.5 fl oz)
- Composition of assembly: Polystyrene (sterilized)

E.1.2.2.3 Filtration procedure.

Select a presterilized filter assembly with a filter of appropriate pore size.

Connect vacuum tubing to the pump and the filter assembly. Use PTFE tubing for pump and filter connections and verify that it is leakproof.

Pour the aqueous sample into the filter funnel portion of the filtration assembly. Avoid excessive turbulence or agitation of the sample, or transferring solids that may have settled to the bottom of the sample container.

Using a vacuum pump, create a negative pressure as recommended by the equipment manufacturer in the collection vessel of the filtration assembly to start the filtration process.

- Collect the filtrate (sample) into the collection flask or other vessel.

Replace the filter funnel portion of the assembly when the filter becomes too restricted because of solids buildup on the filter. To replace the filter, depress the pressure/vacuum release button, disconnect the filter funnel and replace it with a new one, create a vacuum with the hand pump, and continue filtering the remaining sample.

- Release the negative pressure at the vacuum pump and in the filtration equipment; disconnect the collection flask.
- Transfer the filtrate from the collection flask into appropriate sample containers, avoiding excessive turbulence or agitation to the sample.
- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.

Verify that a PTFE liner is present in the cap. Secure the cap tightly.

- Label the sample bottle with an appropriate label. Be sure to include all necessary information.
- Record the information in the field logbook and field sheet, and complete all chain-of-custody documents (see Instruction F-1, "Documentation," Appendix F).
- Discard or decontaminate all sample filtration equipment and materials in accordance with the project sampling and analysis plan.

E.1.3 Potential problems.

E.1.3.1 One inherent problem associated with the filtration of aqueous environmental samples is the filter becoming clogged. The following are some considerations regarding liquid filtration:

- Always have extra filters available at the sampling site.
- Prefilter dirty samples with a larger pore size filter.
- For highly turbid samples a negative filtration system may be more efficient.
- Avoid pouring sediments from the bottom of the collection flask into the filter funnel.
- When the filtrate flow becomes too slow because of filter loading, change the filter. Avoid increasing the pressure and rupturing the filter membrane.

E.1.3.2 To verify the effectiveness of the decontamination procedures, as well as to evaluate the cross-contamination potential of the filter media, recommend collection of equipment blanks. A detailed discussion of equipment blanks is contained in Instruction G-2 (Appendix G).

E.1.4 Other filtration procedures. The filtration techniques outlined in the preceding paragraphs provide some specifics for filtering various liquid media in the field. Other guidelines for filtration techniques exist, i.e., American Society for Testing and Materials (ASTM). As with any technique, the technical team should consider their project objectives and how their procedure will affect the chemistry of the sample before analysis. Such factors as aeration, agitation, temperature, pressure, adsorption, chemical compatibility, etc., should all be considered.

E.2 Homogenizing Techniques

E.2.1 Scope and application. This instruction provides guidance for homogenizing samples. Proper homogenization is vital to accurately assessing the condition of a particular site. Correct homogenization techniques are also important for preparing the necessary quality control (QC) samples associated with a typical sampling event. Homogenization techniques should not be used when samples for volatile organic analyses (VOA) or other parameters that require undisturbed samples are collected.

E.2.2 Sample handling and mixing. An integral part of any sampling investigation is obtaining samples that truly represent the site under investigation. Therefore, applying proper homogenization techniques will help ensure that conditions are being accurately represented. Generation of field control samples (e.g., replicate samples) provides a means for evaluating matrix heterogeneity and the sampling and handling techniques of field personnel. However, for this evaluation to be meaningful, field sampling personnel must be able to properly homogenize and divide collected samples.

E.2.2.1 Sampling equipment composition. The composition of sampling equipment can affect sample analytical results. Sampling materials used must be properly decontaminated and must not contaminate the sample being collected. The standard materials for sampling equipment used to collect samples for trace organic compounds or metals analyses are given in Table E.1. This table may be used as a guide to select the proper sampling instruments.

Table E.1
Standard Materials for Sampling Equipment

Analysis/Site Condition	Preferred Material
Metals	Glass or PTFE
Organics	Stainless steel, glass, or PTFE
Corrosive Soil/Waste	Glass or PTFE

E.2.2.2 Required sample volumes. The volume of sample obtained should be sufficient to perform all required analyses with an additional amount collected to provide for quality control needs, including any split or replicate samples. The volume of sample required by the laboratory depends on the analyses to be performed. Volumes and containers identified in Appendix B are sufficient volumes for the prescribed analysis. If deviations from these volumes are necessary due to low sample yields, the laboratory receiving the sample and conducting the analyses should be consulted for alternative volume requirements. The volumes of samples collected from waste sources at hazardous waste sites or samples from sources that are known to be toxic should be kept to an absolute minimum since disposal costs of excess sample material are high. The laboratory or project personnel may require that excess sample volume be returned to the site because of the hazardous nature of the samples or because of sensitive political issues surrounding the project. If samples are being collected for bench-scale or pilot-scale remediation studies, larger volumes may be necessary. This scenario normally involves sending large bulk volumes to a laboratory to undergo various applications/manipulations to identify the optimum conditions for remediation of a particular waste stream. The data user (i.e., design engineer) or laboratory should be contacted to determine the volume of material required.

E.2.2.3 Aqueous samples. Aqueous samples are typically considered homogeneous because of the physical properties of water, such as diffusion and the ability to flow and freely mix. Therefore, aqueous samples do not require mixing. However, when solids are present within the aqueous samples, viscous or semisolid liquids are encountered, and the sample will require mixing. These samples can be shaken well

or stirred thoroughly with a tool of appropriate composition. The sampler may also encounter portions of the media that are immiscible with water and separate into distinct phases. In these situations, it is advisable to collect a sample from each layer/phase as well as a homogenized sample. When multiple phases are sampled, the sample should be homogenized in the laboratory to achieve the most homogeneous sample. Water samples (potable well, monitoring well, surface water) should be obtained by alternately filling sample containers from the same sampling device for each parameter. Split and replicate samples will be collected simultaneously with the primary samples. Containers for VOA will be filled first, followed by containers for semivolatile organics, metals, cyanide, and water quality parameters. Each VOA container should be completely filled immediately, rather than splitting the water between bottles and filling the bottles incrementally. The containers will all be filled from the sampling device if possible. If this is not possible, a minimum of two containers (one for the primary sample and one for the split sample) will be filled from each sampling volume. If more than two containers can be filled from one sampling volume, the number of containers filled should be an even number (i.e., two or four) so that an equal number of containers for the primary and split samples are prepared. The remaining portions of the sample will then be prepared by splitting each sampling volume between containers for the primary and replicate samples.

E.2.2.4 Solid samples. Obtaining samples in a soil or sediment matrix requires homogenization of the sample aliquot prior to filling sample containers. However, volatile organic samples are the exception; samples being analyzed for volatile organic compounds (VOCs) must always be taken from discrete locations prior to mixing. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOC samples. This practice is necessary to prevent loss of volatile constituents and to preserve, to the extent practicable, the physical integrity of the volatile fraction. Homogenization of the sample for remaining parameters is necessary to create a representative sample media. Moisture content, sediments, and waste materials may inhibit the ability to achieve complete mixing prior to filling sample containers. Consequently, alternative procedures may need to be pursued, i.e., kneading, particle size reduction (PSR), or particle size separation (PSS). However, it is extremely important that solid samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample location.

E.2.2.4.1 Before sample mixing is performed, instructions on the removal of extraneous sample materials (grass or materials in "root zone," leaves, sticks, rocks, etc.) should be given. This can be accomplished by the removal of material by a gloved hand, or through the use of PSS devices (i.e., sieves). Other procedures employed may include PSR techniques. This may be as simple as breaking up large material with a hammer, or may include more elaborate techniques (grinder or mill). However, many of these PSR devices are difficult to decontaminate, and may not be conducive to trace level chemical analyses.

E.2.2.4.2 Homogenization procedures may be accomplished by several methods. The method best suited for the media will depend on the physical characteristics of the solid material (e.g., heterogeneity of media, maximum particle size present, moisture content, etc.). In general, homogenization is accomplished by filling a properly decontaminated container with the sample and mixing it with a decontaminated implement. The container should be large enough to hold the sample volume and accommodate the procedures without spilling. In most cases, the method of choice for mixing is referred to as cone and quartering and can be performed in a bowl or tray of an appropriate material (depending on the analytical parameters to be performed). First all the soils will be disaggregated to less than 6-mm (1/4-in.) diameter as the sample is mixed. The soils are then gathered into a pile in the middle of the container and divided into quarters. Each quarter is mixed, then soils from opposite corners are mixed together again. Soils are then partitioned into quarters again, and this time adjacent corners are mixed together, then the whole combined again. The extent of mixing required will depend on the nature of the sample and should achieve a consistent physical appearance before sample containers are filled. The soils are then divided into final quarters, which are equally subsampled to fill the appropriate containers. If the solid medium is not amenable to cone and

quartering techniques due to the high moisture content or high cohesiveness of the waste, recommend kneading techniques be pursued. First place the sample into a clean noncontaminating bag, and knead materials thoroughly to mix the sample.

E.2.3 Potential problems.

E.2.3.1 The true homogenization of soil, sediment, or sludge samples may be difficult to accomplish under field conditions. However, the homogenizing techniques may be evaluated with the use of a noninterfering dye. The noninterfering dye should be added to the sample medium prior to homogenizing procedures. The resulting distribution of the dye throughout the sample medium during the mixing will indicate the effectiveness of the procedures and areas requiring further mixing.

E.2.3.2 Another important aspect of obtaining a representative sample is to employ proper subsampling techniques. Recommend as a final step of the mixing that the material as a whole be subsampled as equally as possible. This may be accomplished by the procedures already noted or as follows. Flatten the piled material into an oblong shape. Using a flat-bottomed scoop, collect a strip of material across the entire width of the short axis. Repeat this procedure at evenly spaced widths until the sample containers are filled. If the material is cohesive, the solid medium may be flattened, and cut into cubes. Collect random cubes into a subsample, which will be rekneaded and placed into the appropriate sample containers.

E.3 Compositing Samples

E.3.1 Scope and application. This instruction provides information on the various types of composite sampling techniques and the proper procedures to obtain a composite sample. The technique of compositing discrete samples is typically employed when the site under investigation is quite large to improve the precision (lower the variance) of the estimated average contaminant concentrations, especially when contamination exhibits a short-range heterogeneity, and to decrease the probability of making a wrong decision based on limited data. Consultation with data users should be done to determine the appropriateness of applying compositing schemes to meet project objectives. Compositing scenarios that employ a retesting scheme may also be effective in identifying hot spots if a majority of the discrete samples are anticipated to be nondetect and there is adequate sensitivity of the analyses. In this case, the maximum number of discrete samples composited should be determined based on the dilution factor imposed and the sensitivity of the analyses in relation to the project decision level. Compositing schemes are of most benefit when analytical costs are high or analysis is time-consuming relative to sampling costs. Composite sampling may also decrease overall sampling and analytical costs. Composite sampling is not specific to one matrix. Rather it can be utilized for solid, semisolid, liquid, and air matrices.

E.3.2 Compositing techniques. Composite samples consist of a series of discrete grab samples that are mixed together to characterize the average composition of a given material. The discrete samples used to make up a composite sample are typically of equal volume, but may be weighted to reflect an increased flow or volume. Regardless, all discrete samples must be collected in an identical fashion. Likewise, the number of grab samples forming a composite should remain consistent (i.e., a number and pattern for collection of grab samples within a grid should be selected and, for a given grid size, should not be changed). Five types of composite samples are discussed in the following sections.

E.3.2.1 Flow-proportioned composite. Flow-proportional composite samples are collected proportional to the flow rate during the compositing period by either a time-varying/constant volume or a time-constant/varying volume method. This type of sampling is usually associated with wastewater or storm water runoff sampling. To enhance the representativeness of the flow-proportioned composite sample, suggest collection using an automatic sampler that is paced by a flowmeter. Automatic samplers reduce human error, and can directly correlate flow with both sample size and time. Figure E-1a and c illustrate flow-proportioned composite sampling.

E.3.2.2 Time composite. A time composite sample is composed of a varying number of discrete samples collected at equal time intervals during the compositing period. The time composite sample is typically used to sample wastewater and streams, and in some air sampling applications. Time composite samples are typically obtained using automated programmable samplers. When a large number of locations must be sampled, automatic samplers may be set up to sample these locations simultaneously with minimal supervision and costs. In hazardous situations, use of automatic samplers can reduce personnel contact with hazardous waste streams or with potentially dangerous sampling environments. The disadvantages of automatic sampling equipment are its high cost and extensive maintenance requirements. These disadvantages can be offset by reduced labor requirements, proper maintenance, and the proper choice of equipment. When access to the waste stream is relatively easy and sufficient labor is available, manual methods are also quite effective. The most significant disadvantage of manual sampling is that it is labor-intensive, particularly with respect to long-term composite sampling. Figure E-1b illustrates equal time compositing.

E.3.2.3 Areal composite. Areal composite samples are samples collected from individual grab samples collected in an area or on a cross-sectional basis. Areal composites are made up of equal volumes of grab samples where all grabs are collected in an identical manner. Areal composite sampling is typically used for estimating average contaminant concentrations in surface soils or sediments. This is especially

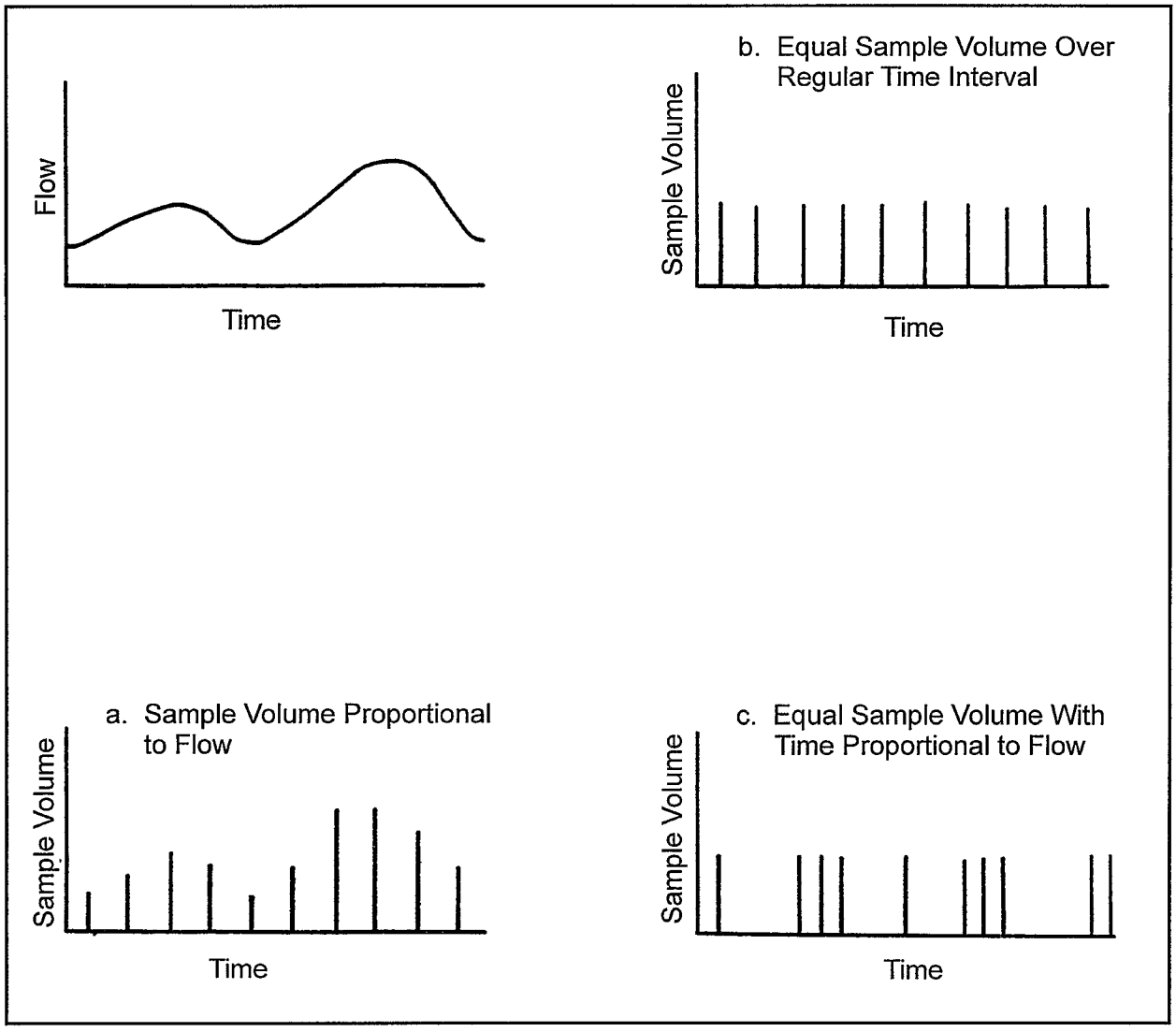

Figure E-1. Composite sampling methods

useful when contaminants are present in a nugget form (i.e., TNT chunks, lead shot, etc.), exhibiting large differences in concentration in a small area (short-range heterogeneity). Grid sizes should be kept moderate (1.5 to 3 m (<5 to 10 ft) in diameter), if project objectives and intended use of the data are to maintain aspects of a "discrete" sample while providing better overall coverage. Reference Jenkins et al. (1996a) for additional details on the use of short range areal composite sampling techniques.

E.3.2.4 Vertical composite. Vertical composite samples are also collected from individual grab samples but taken from a vertical cross section. Vertical composites are also made up of equal volumes of grab samples where all grab samples are collected in an identical manner. Vertical profiles of a soil borehole or sediment columns are examples of vertical compositing.

E.3.2.5 Volume composite. Volume composite samples are collected from discrete samples whose aliquot volumes are proportional to the volume of sampled material. This type of composite is usually

associated with hazardous waste bulking operations, where the composite sample is intended to represent the combined or bulked waste. Discrete samples are typically combined within a group of compatible wastes to undergo physical and chemical testing to define disposal options or determine acceptability at a treatment, storage, and disposal facility.

E.3.3 Compositing grab samples. In general, compositing grab samples lends itself to lowering analytical costs because it reduces the number of analyses. Collecting composite samples also requires project-specific decisions for several key points, including the type of composite sampling technique that will meet the project needs (i.e., time composite, areal composite, etc.); the total number of composite samples needed; the number of grab samples in each composite; and the size and pattern of the sampling grid. These issues may depend on the size of the area under investigation, the nature of the contaminants, and the position of the regulators. Good documentation of sampling locations is also essential in all field sampling, particularly when several grab samples are being homogenized to form a composite. If a contaminant is detected in a composite sample, each of the discrete grab samples that made up the composite should be analyzed individually to determine the actual distribution of the contamination. Procedures should be established between the project manager and the laboratory to ensure that holding times for the discrete grab samples are not exceeded. However, caution should be exercised when reviewing this type of confirmatory analysis due to the lag time between sample analyses and expiration of the holding times of the samples.

E.3.3.1 Solid matrix. Composite samples should be prepared as follows:

- Collect discrete grab samples using the appropriate instructions as outlined in Appendices C and D. To obtain a representative composite sample, it is important that all grab samples are collected in identical fashion.

- Homogenize the individual discrete samples as outlined in Instruction E-2, and place them into properly labeled sample containers.

- Assemble the sample containers that contain the grab samples that will make up a specific composite sample.

- Remove an appropriate volume of discrete sample (aliquot) from each sample container and place it into a clean stainless steel mixing bowl. Each aliquot amount should be taken in an identical fashion to facilitate representativeness. Avoid generating excess contaminated soil when possible.

- Homogenize the aliquots as described in Instruction E-2.

- Remove sample amounts from the homogenized composite sample and place them into the proper containers for shipment to the laboratory.

- Place the individual homogenized discrete samples in proper storage conditions after aliquots are removed for compositing, when a retesting scheme is employed, or if it is of benefit to the project. If the composite sample results do not appear to be accurate or if evidence of contamination exists, subsequent analyses of the individual grab samples that composed the composite may confirm the results and provide discrete information.

E.3.3.2 Liquid matrix. The preparation of liquid matrix composite samples is typically easier than that of solid matrices due to the tendency of liquids to homogenize easily. Also, it is common practice to send liquid grab samples to the laboratory for compositing because of the difficulties in handling larger sample volumes (4 to 16 L (1 to 4 gal) for a typical wastewater sampling event) and to minimize the potential

to introduce contaminants. When liquid composite samples are to be generated in the field, the following procedure should be used:

- Assemble all sample containers that contain the grab samples that will make up a specific composite sample.
- Shake or stir the individual containers to homogenize.
- Using clean glass or disposable pipets, deliver aliquots of the homogenized grab samples directly into a sample container to be sent to the laboratory. (It will require five 200-mL (7-fl-oz) aliquots from five discrete grab samples to generate a 1,000-mL (33-fl-oz) composite sample).
- Seal the container and shake well to mix. Avoid stirring samples if possible to lower the potential of introducing contaminants.
- At some sites it may be beneficial to save and store the individual homogenized grab samples after aliquots are removed for compositing. If the composite sample results do not appear to be accurate, subsequent analyses of the individual grab samples that composed the composite may confirm the results. Confirmatory analyses of these samples would likely be for informational purposes only since the holding times of the samples may have expired.

E.3.4 Potential problems.

E.3.4.1 Compositing does not allow the spatial variability of contamination or discrete information to be determined. Additional analyses of the individual grab samples are required.

E.3.4.2 Low concentrations of contaminants in individual grab samples may be diluted so that the total composite concentration is below the detection limit. In this case, the existence of the contamination in individual samples would go unnoticed. Therefore, the maximum number of discrete samples composited should be based on the dilution factor from the compositing and the analytical sensitivity in comparison to the project decision level and sensitivity requirements.

E.3.4.3 When the sampled medium is not amenable to mixing techniques (samples are moist and clayey), it may be very difficult to create a homogeneous sample mixture. Consequently, the resulting composite may not represent an average of all the grabs.

E.3.4.4 Compositing techniques should not be employed when chemical interactions may diminish the integrity of the sample (i.e., VOC samples).

E.3.4.5 Compositing schemes are not efficient when the goal is to identify hot spots and there is a high probability that the discrete samples contain detectable concentrations. The amount of retesting may be significant to achieve the objectives.

E.3.4.6 Compositing schemes are not efficient if analytical costs are low.

E.3.4.7 Obtaining samples by an automatic sampling device is typically difficult for the first- time user. However, after the sampler has become familiar with the sampling device and any problems have been addressed, these devices prove to be quite reliable.

E.4 Collection, Handling, and Storage of Solid Samples for VOC Analysis

E.4.1 Scope and application.

E.4.1.1 This instruction presents guidance for the collection and handling of surface/subsurface sediments, soils, and solid hazardous waste materials taken for VOC characterization. The procedures include collection, handling, storage, and onsite preparation for analysis of discrete samples; and collection, handling, and storage of discrete samples for offsite sample preparation. Information concerning the selection and application of the sampling devices available for subsurface bulk sample collection can be found in Instructions C-5 and C-6, Appendix C, and Instruction D-1, Appendix D.

E.4.1.2 Special procedures are necessary for VOCs, since in most solid matrices these analytes coexist in gaseous, liquid, and solid (sorbed) phases. Loss of analyte from any one phase may render the sample unrepresentative of the material as a whole. Therefore, sample collection, handling, and analysis must be performed under conditions that maintain the accountability of VOCs in all phases. In general, uncontrolled VOC losses occur through two mechanisms: volatilization and degradation. Volatilization losses occur whenever gaseous molecules are allowed to move away freely. This loss mechanism usually dominates whenever a new surface is created. Traditional sampling procedures used for acquisition of solid VOC samples are very susceptible to this type of loss. Further losses during transport and storage are common if fine soil grains are present on the threads of the vial or at sealing surfaces, thereby preventing a good seal. The most significant VOC losses, however, are due to the reopening of the vial and sample handling at the laboratory. In general, the extent to which VOCs are lost will depend on the vapor phase concentration (analyte vapor pressure), extent of surface area exposure, length of exposure, and porosity of the sample matrix. However, studies have shown VOC losses of a magnitude 10X and higher are common. Degradation losses are usually attributable to biological processes. Aerobic biological degradation dominates, because traditional intrusive collection methods expose the sample to oxygen in the atmosphere. The rate of biological degradation depends on several factors, including the indigenous microbiological population, chemical properties of the VOC, nutrients, moisture, and temperature. Aromatic compounds are quite susceptible to this loss mechanism, and preservation by cooling to 4°C ± 2°C has been found to be insufficient in retarding biological degradation.

E.4.1.3 Solid sample preparation for VOC analysis is typically performed by vapor partitioning (i.e., purge-and-trap or headspace), or by methanol extraction. Refer to Method 5035, EPA/SW-846, for these sample collection procedures. In general, when VOC analysis is performed by gas chromatography or gas chromatography/mass spectrometry, vapor partitioning methods are used for solid samples thought to be contaminated with VOCs at levels lower than 0.2 mg/kg (Method 5035, low-level method). In contrast, solid samples thought to have concentrations above 0.2 mg/kg are analyzed after extraction (dilution) with methanol (Method 5035, high-level method). Method 5035 has been designed to improve the sample handling and preservation procedures and minimize the negative bias in VOC results by incorporating several preparatory steps traditionally performed in the laboratory into the field. These sample collection procedures differ significantly from traditional methods and impact several technical personnel in both the field and laboratory. These changes require increased coordination and communication between parties involved to ensure successful acquisition of representative solid VOC samples. The procedures discussed in this instruction are for clarification and implementation of SW-846 Method 5035, and are designed to limit sample VOC losses by volatilization and biodegradation. This is accomplished by stressing that samples are collected only from freshly exposed surfaces, sample collection and transfer are performed quickly and in a nondisruptive fashion whenever possible, sample procedures follow Method 5035 low-level (purge and trap), or high-level (methanol) extraction procedures, or samples are taken in an airtight vessel cooled to 4°C ± 2°C, and are held up to only 48 hours prior to analysis. It is important to recognize that Method 5035 low-level (closed-system purge-and-trap) procedure requires the laboratory to have special equipment

designed to handle VOA vials in an automated sample introduction system. Furthermore, these methods of analysis do not necessarily apply to water-soluble VOCs. For information concerning the analysis of water-soluble VOCs, refer to SW-846 Method 5000.

E.4.2 Sampling strategy and number of samples.

E.4.2.1 In general, the selection of methodology — low-level versus high-level method — will depend on project data quality objectives (DQOs) (action or decision levels), and the expected VOC concentrations of the environmental matrices to be sampled. This is illustrated in the flow chart in Figure E-2. As shown in Figure E-2, the high-level method is used when VOC action levels are relatively high or the VOC concentrations are greater than 200 g/kg. The low-level method is used when project action levels are low, or site VOC concentrations are less than 200 g/kg. When action levels are low, field screening should be performed to direct the acquisition of either low-level or high-level samples. Recommend the field screening be performed by onsite gas chromotography, or according to procedures described in Hewitt and Lukash 1997. If no field information is available, both low-level and high-level samples must be collected. The collection of both low-level and high-level samples for fixed-laboratory analyses constitutes the most conservative approach to avoid the need for remobilization/resampling efforts to obtain necessary data.

E.4.2.2 Screening techniques at the laboratory are also recommended to confirm any onsite results and to avoid damage to laboratory instrumentation. If no onsite or laboratory screening is performed, and both low-level and high-level samples are submitted to the laboratory, the laboratory should perform the high-level analyses first, and if no VOCs are detected, analyze the corresponding low-level sample. If the low-level sample is analyzed initially without further information, the laboratory runs the risk of contaminating the analytical system (requiring significant maintenance) and potentially impacting data of other samples within that analytical batch. Reanalysis using appropriate preparatory procedures is necessary for any samples that exceed the calibration range of the instrument.

E.4.2.3 Regardless of the methodology employed, several collocated samples will generally be required for each sample location (e.g., from each sampling depth or soil boring). The exact number of required collocated samples will depend on several factors, including analytical methodology (the high-level versus the low-level method), field screening results, the laboratory's protocols for screening of samples, and project requirements for field QC samples (e.g., matrix spikes and duplicates). For example, when low-level analysis is required and field screening results show site VOC concentrations to be low, at least two samples must be collected for analysis. Two samples are necessary due to the entire vial being processed during the VOC analysis. The second vial allows the laboratory an opportunity to perform an additional low-level analysis should the first analysis be unacceptable. When low-level analysis is required and the site VOC concentrations are unknown, at least two samples must be collected for potential low-level analysis and one sample must be collected for potential high-level analysis. The high-level sample is subsampled and the aliquot of methanol extract diluted for VOC analysis. Therefore, one high-level sample can accommodate multiple high-level analyses. Finally, if the laboratory plans to screen the samples, an aliquot of the high-level sample may be used, or an additional sample may be collected.

E.4.2.4 For acquisition of QC samples, field screening information becomes even more important. In order to avoid the need to collect both low-level and high-level QC samples, field screening should be performed, or alternative collection procedures (i.e., EnCore™) employed for these QC samples. For example, the field duplicate typically requires one additional collocated sample, while the matrix spike/matrix spike duplicate requires an additional two samples. However, if no information on site VOC concentrations is available, this could expand to three samples for the field duplicate (two for low-level and one for high-level), and six additional samples for the matrix spike/matrix spike duplicate. In addition to the samples

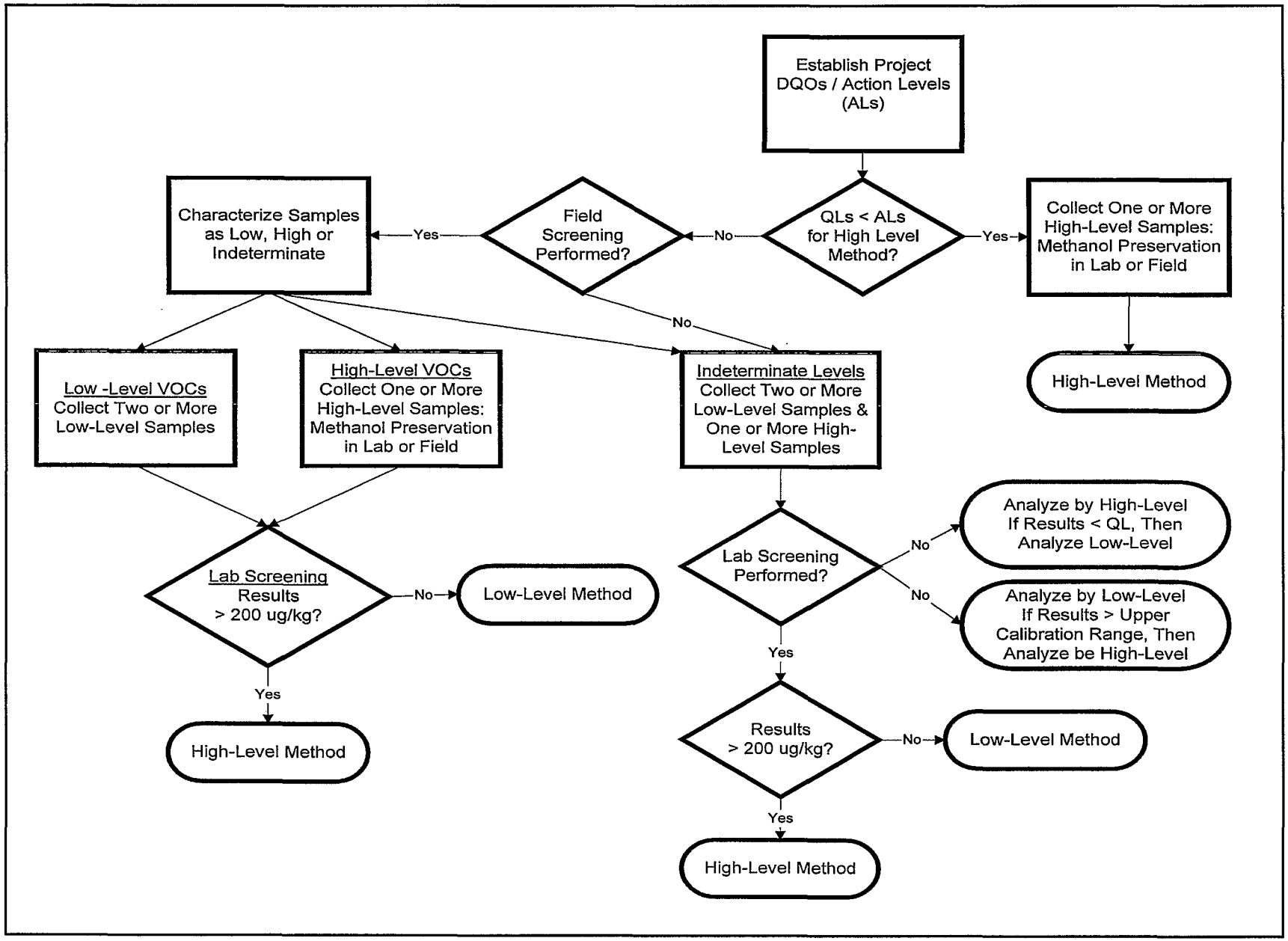

Figure E-2. VOC analysis decision tree

collected for VOC analysis, another collocated sample must be collected for a moisture content determination in order to report the VOC results on a dry-weight basis. Samples for moisture content determinations may be collected in conventional VOA vials and cooled to 4 °C ± 2 °C. Proper project coordination between field and laboratory personnel and implementation of onsite and/or laboratory screening processes can help reduce the numbers of samples to manageable quantities.

E.4.3 Sample collection summary. In order to minimize VOC losses, Method 5035 sample collection and preparation procedures dramatically modify both the low-level and high-level VOC methods. The revised sample collection techniques greatly reduce the time in which samples are exposed to atmospheric conditions. Initially to help maintain the physical structure of samples of a cohesive granular material, a hand-operated coring device must be used to collect the appropriate sample size for laboratory analysis (e.g., cylindrical soil columns are extruded into vials using disposable plastic syringes with the tapered front ends removed). However, some materials (e.g., cemented or noncohesive granular material) may be too difficult for coring tools to penetrate or contain. These materials can be sampled by fragmenting a larger portion of the material with a clean chisel to generate aggregate(s) of a size that can follow sampling protocol 2 (placed into a VOA vial or bottle containing chemical preservative). When aggregate(s) are transferred, precautions must be taken to prevent compromising the sealing surfaces and threads of the container. Losses of VOCs using this procedure are dependent on the location of the contaminant relative to the surface of the material being sampled. Therefore, caution should be used during data interpretation. As a last resort when this task cannot be performed onsite, a large consolidated sample can be collected in a vaportight container and transported to the laboratory for subsampling. Sample protocol 2 presents a sample being added to collection vials containing chemical preservatives such as sodium bisulfate solution or methanol for the low-level and high-level methods, respectively. Field personnel transfer samples immediately into preweighed vials containing chemical preservatives without additional sample handling. The vials and chemical preservative are weighed in the field before use, and are reweighed after the sample aliquot addition to obtain the net sample weight. Alternatively, samples for both the low-level and high-level methods may be collected following sample protocol 1. Sampling is performed with a coring device, which becomes the sample container and is hermetically sealed (e.g., EnCore™ sampler from En Chem, Inc.) and stored at 4 °C ± 2 °C for a maximum of 48 hours.

E.4.3.1 Sampling protocol 1.

E.4.3.1.1 Sampling protocol 1 consists of a coring device that also serves as a shipping container. Presently, the EnCore™ sampler is the only commercially available coring device that was designed to collect, store, and transfer soils with minimal loss of VOCs. The disposable EnCore™ sampler is designed to be a single-use coring device that stores the soil sample in a hermetically sealed, headspace-free containment that maintains sample integrity. Most soils that require sampling will consist of cohesive granular materials, which allow the use of such a coring device. However, the sampling protocol will not be applicable to all solid environmental matrices. Some geological materials are impossible to core (e.g., gravels and hard dry clays). Refer to sampling protocol 2 for guidance on handling these materials. The EnCore™ sampler has a hand-operated coring tool available for obtaining 5-g and 25-g samples. The 25-g sampler is designed for the zero headspace extraction for purposes of the VOC Toxicity Characteristic Leaching Procedure testing. Note that the 25-g sampler should not be used to collect, store, and transfer soils from the field for subsampling in the laboratory into 5-g aliquots. This additional sample handling would defeat the benefit that the EnCore™ sampler affords.

E.4.3.1.2 Advantages of sampling protocol 1 include the simplified field procedures, which do not require sample weighing or addition of preservatives in the field. Because sample preparation is performed at the laboratory, exposure hazards and Department of Transportation (DOT) shipping issues arising from

the field application of chemical preservatives such as methanol are also avoided. However, samples must be stored at 4 °C ± 2 °C and prepared for analysis within 48 hours of collection. The short holding time for sample preparation usually requires additional coordination with the analytical laboratory and may involve higher analytical costs. The following is general guidance for the collection of a soil sample using the EnCore™ sampler:

- After the split spoon is opened and a fresh surface is exposed to the atmosphere, the sample collection process should be completed in a minimal amount of time with the least amount of disruption (disaggregation) as possible. Visual inspection and an appropriate screening method (e.g., photoionization detector or flame ionization detector readings) may be selected to determine the interval of the soil core to be sampled.

- Rough trimming of the sampling location surface layers should be considered if the material may have already lost VOCs (been exposed for more than a few minutes) or if it may be contaminated by other waste, different soil strata, or vegetation. Surface layers can be removed by scraping the surface using a clean spatula, scoop, or knife.

- Insert the clean coring tool into a fresh surface for sample collection. Take care not to trap air behind the sample. An undisturbed sample is obtained by pushing the barrel of the coring tool into a freshly exposed surface and removing the corer once it is filled.

- The exterior of the barrel should be quickly wiped with a clean disposable towel to ensure a tight seal and the cap snapped on the open end.

- The sampler should be labeled, inserted into the sealable pouch, and immediately cooled to 4 °C ± 2 °C.

- Repeat this procedure to collect separate collocated samples for moisture content and any QC samples.

- Prepare the shipment to go to the laboratory. If samples are going to be shipped near the weekend or holiday, recommend coordinating with the receiving laboratory to ensure holding time of 48 hours for the EnCore™ sample is met.

E.4.3.2 Sampling protocol 2.

E.4.3.2.1 Sampling protocol 2 is applicable to all solid matrices. However, if soils are not retained within the coring device (i.e., wet enough to flow), it may be necessary to cover the open end of the coring device with aluminum foil in a manner that will maintain sample integrity until the material is transferred to appropriate sample vials. When gravel, or a mixture of gravel and fines, that cannot be easily obtained using coring tools is sampled, a sample may have to be transferred using a clean spatula or scoop. The collection vial will contain the chemical preservative; therefore, samples should be dislodged with minimal splashing and without the spatula or scoop contacting the liquid contents. For some solids, a wide-bottom funnel or similar channeling device may be necessary to facilitate transfer to the container and prevent compromising the sealing surfaces of the container. Losses of VOCs are likely because of the additional sample handling and the noncohesive nature of the material, which exposes more surface area to the atmosphere. Another potential source of error during the subsampling process is the separation of coarser materials from fines, which can skew the concentration data if the VOC contamination is associated with particular particle sizes, which are not properly represented in the sample. Also, due to an aqueous acidic

solution of sodium bisulfate being used to preserve samples for the low-level analyses, samples must be tested for carbonate interferences in the field before the samples are containerized. If carbonates are present, the sodium bisulfate may potentially react with the carbonates producing effervescence, which promotes the loss of VOCs. The high-level method is not affected by the presence of carbonates. In these cases, alternative procedures (i.e., EnCore™ or high-level method) should be used. All sampling difficulties should be well documented in field logbooks and caution used in the interpretation of the data obtained from these types of materials. Finally, recommend the field personnel become familiar with the volume of material needed to reach the estimated 5 (or project-specific) grams. For cohesive soils, recommend approximating a mark on the disposable syringes to help guide the acquisition of the soils needed. For other materials, preweighing materials to the volume needed, which are then discarded, will help visualize the amount of material to be transferred when sampling time and sample handling are critical.

E.4.3.2.2 All of the sample containers used should be made of glass and have a thick septum cushion between the sealing material (PTFE) liner and cap (rigid plastic screw cap or aluminum crimp top). PTFE-lined caps for bottles should have flexible septum backing and be at least 10 mils thick to ensure a liquid or airtight seal. The sample containers (VOA vials) containing appropriate volume and grade (e.g., purge-and-trap grade methanol) chemical preservative should be prepared by the laboratory prior to shipment to the field. Surrogate compound(s) may be added by the laboratory at this time to allow an assessment of the sampling procedures. The laboratory should be responsible for providing trip blanks and ambient blanks (e.g., methanol). Note that the sample vials for the Method 5035 low-level method are designed to be placed directly on the laboratory's instrument (i.e., auto sampler) so that they remain within the closed system up to and during VOCs analysis. Therefore, it is critical that only the 40-mL VOA vial (and not the 60-mL VOA vial) containing the magnetic stir bars be used for the low-level analysis. Recommend that disposable stir bars be used since memory effects have been reported with magnetic stir bars that have been reused without effective decontamination. Recommend the laboratory note the tare weight of the sample vial with preservative (and stir bar, if necessary) on the sample label before sample vial shipment. After this initial weighing, sample containers should be opened only to transfer sample into them. After the sample transfer into the collection vessel, the sample container is reweighed in the field (and again in the laboratory prior to analyses). The difference in the weight, measured before and after the sample is introduced, is used to establish the sample wet weight. Any discrepancies between field weights and laboratory weights must be thoroughly documented to assess the loss of sample or extract and the acceptability of the sample for analysis as outlined in Section E.4.4.1. The following is general guidance for the collection of soil samples for field preservation for Method 5035 low-level or high-level methods.

- Solid matrices should be screened for carbonates. Refer to Section E.4.3.2.1 if carbonates are present.

- Field personnel should record the weight of the sample vials containing preservative to verify consistency with the laboratory tare weight and ensure no loss during initial transport of the sample containers to the field. Note any discrepancies back to the laboratory and in the field logbook.

- The coring device should be prepared as follows: cut off tapered front end of a disposable plastic syringe and remove the rubber cap from the plunger. A mark may be placed on the syringe to approximate the volume of material needed.

- After the split spoon is opened and a fresh surface is exposed to the atmosphere, the sample collection process should be completed in a minimal amount of time with the least amount of disruption (disaggregation) as possible. Visual inspection and an appropriate screening method

(e.g., photoionization detector or flame ionization detector readings) may be selected to determine the interval of the soil core to be sampled.

- Rough trimming of the sampling location surface layers should be considered if the material may have already lost VOCs (been exposed for more than a few minutes) or if it may be contaminated by other waste, different soil strata, or vegetation. Surface layers can be removed by scraping the surface using a clean spatula, scoop, or knife.

- Insert the clean coring tool (e.g., prepared disposable syringe) into a fresh surface for sample collection. Take care not to trap air behind the sample. An undisturbed sample is obtained by pushing the barrel of the coring tool into a freshly exposed surface and removing the corer once it is filled.

- Hold the vial or bottle containing chemical preservative at an angle when extruding the sample into the container to minimize splashing.

- Perform a visual inspection of the lip and threads of the sample vessel. Remove any foreign debris with a clean towel and cap the vial.

- Tap the vial gently while holding it in an upright position. The purpose of the agitation is to ensure that the preservative completely contacts the soil surfaces and disaggregate any large clumps. The sample vials should not be shaken vigorously or up and down.

- Measure and record the weight of each container into the field logbook and in documentation to the laboratory. Calculate the difference in weight of the container, measured before and after the sample is added, and use to determine the sample wet weight.

- Each of the samples should be immediately placed into smaller sealable plastic bags, collected within a larger plastic bag, placed inside a cooler in an upright position, and cooled to 4 °C ± 2 °C. Because of packaging constraints for shipping (e.g., need for inner receptacles), it is absolutely critical that samples be prechilled to 4 °C ± 2 °C prior to shipment.

- Repeat these procedures to collect separate collocated samples for moisture content and any QC samples.

- The samples are then prepared for shipment to the laboratory following the criteria and regulatory considerations described in Instruction F-2 of Appendix F.

E.4.4 Potential problems.

E.4.4.1 Field weighing. When field personnel collect samples using sampling protocol 2, they essentially perform the following activities for both the low- and high-level methods. Field personnel must weigh the vials containing the chemical preservatives (e.g., aqueous sodium bisulfate for the low-level and methanol for the high-level method), collect the samples using some type of coring device (e.g., a syringe with its tip removed), extrude the sample cores into the vials, and reweigh the filled vials (to determine the sample wet weight for analysis). A net sample weight of about 5 g is required (assuming a soil density of 1.7 g/cm^3, this corresponds to a soil volume of about 3 cm^3). According to Method 5035, weights may be made in the laboratory and/or field. If weights are recorded in both locations, this information may be used to track any loss of sample or preservative. If field weight measurements are used, a loss of up to 0.2 g is

allowed for the vial to be considered acceptable for sample analysis. To the extent possible under field conditions, sample containers should be weighed and samples collected in a "protected" environment to permit accurate weighing and handling. Weights should be recorded to the nearest 0.1 g (or 0.01 g if balance allows) for both the low-level and high-level samples. In addition, the meniscus of the chemical preservative may be marked on the sample container to aid in the evaluation of evaporation, accidental spillage in the field, or loss during shipment. Any sample container that shows a loss of methanol (e.g., meniscus below the line marked by the lab) should be discarded.

E.4.4.2 Chemical interactions. Although not substantiated, there have been two occurrences with methanol and sodium bisulfate preservation that require discussion. In the first case, soils that contain aluminum silicates may act as a catalyst causing the conversion of methanol to acetone. The possible mechanism for this interaction is being researched. In the second case, soils like lignite or peat contain a polymeric constituent known as humic acid that may also interact with sodium bisulfate to form acetone. Until either of these two mechanisms can be confirmed or denied, projects should evaluate the potential for acetone to be a site contaminant. For example, if acetone is not an analyte of concern, then the issue may not impact project decisions. However, those projects that cannot remove acetone from the analyte list should be aware of these possible interactions and any acetone detects should be evaluated. A logical source of acetone contamination is the laboratory. Therefore, site-specific sources should always be assessed and not necessarily attributed to one of these interactions.

E.4.4.3 Shipping concerns. DOT shipping requirements need to be taken into account for the preservatives used. Depending on the quantity and method of packaging, sodium bisulfate and methanol may be DOT hazardous materials and subject to DOT hazardous materials regulations. Refer to Instruction F-2, Appendix F, for additional information.

E.4.4.4 Site safety concerns. Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Protective gloves should be worn when vials containing methanol are handled. Methanol should be stored away from open flames, areas of extreme heat, and other ignition sources. Vials containing methanol should be refrigerated (e.g., stored in coolers with ice). Sodium bisulfate is a strong mineral acid and must be handled with all safety precautions related to acids. Contact with the skin and eyes should be avoided. Protective gloves and eye protection should be worn with vials containing sodium bisulfate.

E.4.4.5 Preservative. When samples are preserved with methanol in the field, it is especially critical to avoid the introduction of contamination from external sources such as vehicular emissions or dust. Hence, when samples are preserved with methanol in the field, a methanol blank should be exposed to field conditions during the sample collection process.

E.4.4.6 Boiling point. Sampling protocol 1 using the EnCore™ sampler has not been demonstrated for compounds with boiling points less than 30 °C (e.g. bromomethane, chloroethane, chloromethane, or vinyl chloride).

E.4.4.7 Costs. Significant analytical costs may be incurred due to the level of redundant analyses (both high-level and low-level) when no site VOC concentrations are known. Recommend implementation of screening procedures at the field and/or laboratory to reduce these costs.

E.5 Laboratory Subsampling

E.5.1 Scope and application. This instruction provides direction on how to obtain a representative aliquot of sample for laboratory analysis. Obtaining this aliquot is referred to as laboratory subsampling and is performed by laboratory personnel during sample preparation steps. Current SW-846 and other standard reference methods provide little or no guidance within their preparatory methods for these critical procedures. For this reason, this instruction addresses subsampling techniques for solid, liquid, and multiphased matrices to be used by all laboratories to ensure consistency in subsampling techniques and in the resulting data. It should be noted that specific samples may require special techniques due to problematic matrices or project- specific requirements. Project-specific guidance should be obtained from appropriate project documentation (i.e., Quality Assurance Project Plan) or technical personnel. Good analytical techniques are also required during all subsampling procedures to obtain representative subsample aliquots, minimize potential bias, and accurately assess any contamination at the site. Procedures for the acquisition of soil samples for VOA sample analysis are not included here. Refer to Instruction E-4 of this appendix for information on these procedures.

E.5.2 Subsampling procedures. It is common for many analytical methods to require only a portion of the submitted sample to be subjected to the actual analysis. Excess sample volume is desirable when there is a potential that the sample will need to be reanalyzed. Because only a portion of the submitted sample is actually involved in the evaluation of the sampling location, it is important that the subsample be truly representative of the entire sample submitted. In general, subsampling techniques are distinguished by the analytical method requirements, the distribution of the contaminant within the sampled medium, and the state or condition of the aliquot to be tested. Information covering these topics are routinely available to the laboratory for all except for the contaminants distribution within the evaluated media. Therefore, unless information exists to the contrary, the laboratory should assume the contamination is distributed throughout the laboratory samples. Due to the impact of each sample on the procedures used, recommend that subsampling procedures be continuously reevaluated based on the individual matrix under assessment, subsequent analysis to be performed, and the intended use of resulting data (if known). When project DQOs dictate alternative procedures to those outlined in the following subsections, recommend project subsampling instructions be submitted along with samples to outline proper procedures to be employed. If no project instructions are provided, the following guidance should be used to establish appropriate subsampling practices. Environmental samples, which are not considered undisturbed, should be homogenized prior to arrival at the laboratory. However, laboratory personnel should not assume these samples are properly homogenized in the field. In addition, both solid and liquid matrices experience settling or phase separation during transport. Therefore, it is critical that submitted samples be properly homogenized prior to subsampling when appropriate for the analysis. Techniques used to homogenize or rehomogenize samples should be documented and executed in accordance with the guidance presented in Instruction E-2. Initial inspection of each sample to determine the sample phases, such as liquid, solid, or a combination (multiphasic), is critical. Laboratory personnel should document the physical appearance of samples upon receipt, including comments about settling and phase separation. Based on the outcome of this assessment, the following procedures should be used to outline general guidelines for subsampling a variety of matrices.

E.5.2.1 Solid matrix. Soil, sediment, homogeneous and heterogeneous solid substances, concrete, paint chips, ash, etc., are to be subsampled for nonvolatile analyses according to the following guidelines. Procedures for the acquisition of soil samples for VOA sample analysis are not addressed. Refer to Instruction E-4 for information on these procedures. When solid matrices are subsampled, a decision must be made as to whether a representative subsample can be obtained without prior sample manipulation. This depends on whether the sample is homogeneous or heterogeneous in nature, as determined by visual inspection of the physical attributes of the sample, and on determining the sample particle size distribution. Particle size is

the physical dimension of the individual parts (i.e., grains of soil) of the sample. Then consider the following questions:

Is there a significant amount of oversized material (be it either naturally occurring (rocks) or artificially introduced material (debris))? Is this material intended to be included/excluded?

Are the contaminants present on a molecular scale or macroscale? For instance, are there obvious chemical inclusions (e.g., lead shot, metal chunks, tar balls, solid chemical material (grayish-white explosives)) indicating a macroscale contamination, or is contamination due to a spill/discharge that adsorbed onto individual soil particles indicating a molecular- scale contamination. Contamination on a macroscale is more susceptible to bias during subsampling procedures. Therefore, project-specific instructions (e.g., compositing scheme) should be formulated based on the purpose of the data. Refer to Instruction E-3 for additional information on this application.

Does the sample tend to segregate into various size fractions easily? Can they be mixed to produce an even distribution prior to subsampling, or do these fractions need to be physically separated and subsampled individually for recombination into an appropriate sample aliquot?

Does the sample maximum particle size meet the minimum allowable class size (as measured by U.S. Standard sieve mesh) as noted in Table E-2, as determined by the subsample mass taken?

Table E-2 Maximum Particle Size Allowed for Subsampling Solid Materials

Subsample Mass (g)	Maximum Particle Size Allowed (cm)	U.S. Standard Sieve Mesh	Wentworth Size Class
0.5 - 1	0.1	18	Coarse sand
2 - 5	0.17	12	Very coarse sand
10	0.21	10	Granule gravel
30	0.31	7	Granule gravel
50	0.37	6	Granule gravel
100	0.46	5	Pebble gravel

If conditions indicate that the sample medium is not homogeneous or that a problem may exist, additional measures may be necessary to obtain a representative sample. These may include techniques for PSR, PSS, sample homogenization, or increasing the size of the sample aliquot taken for analysis. Determining what procedures should be employed may require communication with the data user. Another important aspect of subsampling depends on the method of analysis and the size of the aliquot being taken. Analysis requiring smaller (1-2 g) aliquots (metals, explosives, etc.) requires special considerations so as not to bias the small sample size compared with analysis requiring larger (30 g, extractable organics, or 100 g, toxicity characteristic leaching procedure) analyses. In general, PSR is necessary when the maximum particle size present within the sample is larger than the recommended maximum particle size based on the mass (amount) of the sample aliquot taken for analysis. PSS techniques (sieving) should be performed after PSR, to ensure the desired particle size has been achieved. If PSR (i.e., grinding or milling) or PSS (i.e., sieving) techniques are used, care should be taken to implement the appropriate quality controls, i.e., appropriate inspection and decontamination of devices, to ensure that samples are not contaminated or cross-contaminated during their use.

E.5.2.1.1 Procedures for subsampling homogeneous materials for nonvolatile analyses.

Allow the sample and container to equilibrate to room temperature before opening the container.

Visually inspect and document the appearance of the sample prior to subsampling.

Samples received in brass liners or sections of brass liners must first be extruded onto laboratory tray or pan and mixed as described in the following paragraph.

Even if the material received appears homogeneous, the entire sample should be thoroughly mixed using an inert, noncontaminating spatula or rod material by the procedures outlined in Instruction E-2. This may be performed within the original sample container. However, it is more effective and recommended that the material be transferred onto a laboratory tray or pan.

For cohesive material, the bulk material should first be reduced in size using a stiff- bladed utensil such that the average size of any clump is approximately pea-sized (approximately 6 mm). If the solid medium is not amenable to these techniques due to the high moisture content or high cohesiveness of the waste, recommend using kneading techniques presented in Instruction E-2.

To prepare the subsample, flatten the piled material into an oblong shape. Using a flat- bottomed scoop, collect a strip of material across the entire width of the short axis. Repeat this procedure at evenly spaced widths until the sample aliquot needed is obtained.

- Alternatively, the sample should be subdivided (quartered) and approximately equal portions removed from each quarter of the sample for inclusion into a final sample aliquot that will be accurately weighed into a clean glass beaker or similar container and analyzed.

If the material is cohesive, the solid medium may be flattened and cut into cubes. Collect random cubes into a subsample that will be rekneaded and placed into the appropriate sample containers.

E.5.2.1.2 Procedures for subsampling heterogeneous materials for nonvolatile analyses.

With heterogeneous material a decision must be made as to how a subsample is to be taken so project needs are met. This decision must be made in conjunction with the project personnel who submitted the samples for analysis. The following options are possible solutions in producing a representative subsample of a heterogeneous material.

Achieving a representative subsample must consider whether PSR or PSS techniques are required. If PSR or PSS techniques are used, implement the appropriate quality controls to ensure that samples are not contaminated or cross-contaminated.

PSR should be avoided for semivolatile organic procedures due to the potential loss of more volatile analytes. Other general considerations include contamination and cross-contamination that may result from the devices used during these techniques. If PSR is necessary, mortar and pestle is usually the best alternative for semivolatile methods. Milling is also an effective technique, but samples must be dry or dried, therefore less desirable for semivolatile methods.

If PSR techniques are used that require dry samples, they should be dried at room temperature to a constant weight without exposure to direct sunlight or heat.

If the sample is amenable to PSR (e.g., grinding or milling), refer to general guidance presented previously and process the entire sample in an appropriate device (meaning that target analytes are unaffected by the use of the particular grinding apparatus).

After PSR, use appropriate sieve (PSS) to check for oversized material.

Do not rework the oversized material. Add more original sample to the PSR equipment and repeat procedures until a sufficient amount of material is generated.

Following PSR/PSS techniques, mix the sample until homogeneous and subsample as outlined previously.

If the heterogeneity is due to foreign material or debris (and project DQOs allow), physically separate the foreign material, mix the remaining homogeneous sample, and transfer an appropriately homogeneous sample to an appropriately sized aliquot to the tared testing vessel(s).

Or cone and quarter the samples as outlined in Instruction E-2, repeating this procedure until the subsample obtained meets the sample size of the analytical procedure.

For samples that segregate into size fractions easily, perform a PSS procedure (e.g., sieving).

Determine the percentage of each size fraction.

Compose a subsample that takes an appropriate percentage of each fraction.

E.5.2.2 Aqueous liquid matrix. Samples of surface water, ground water, toxicity characteristic leaching procedure extracts, wastewaters, and leachates containing <1 percent solids are sampled using the following guidelines. Evaluate the liquid sample, looking for suspended matter, multiple phases, or any other features that may require specific measures to obtain a representative subsample. This may be restricted due to the container material (i.e., amber glass). If, upon inspection, it is discovered that the sample has more than one liquid phase, or greater than 10 percent of the sample is sediment or solid fines, consult with the project technical personnel to determine sampling needs.

E.5.2.2.1 For aqueous liquids that are to be analyzed for inorganics and total metals:

Allow the sample and container to equilibrate to room temperature.

Secure the sample lid to sample jar. Then invert, or shake the sample up and down a minimum of three times.

Evaluate whether the rate that the suspended matter settles allows sufficient time to acquire a representative aliquot.

If the suspended matter settles slowly, shake the sample repeatedly and take an aliquot immediately following procedures outlined in the following procedures.

If the suspended matter settles rapidly, shake the sample repeatedly and immediately transfer to a large beaker. Add a magnetic stir bar and magnetically stir the sample until uniformly mixed. While stirring continues, take an aliquot by pipet or other subsampling means.

Remove lid and quickly yet smoothly transfer desired aliquot into an appropriately sized graduated cylinder or volumetric flask. This choice will be dependent on the required accuracy necessary for the measurement device as specified in the applicable method. If the required sample volume is small, a volumetric pipet may be used to obtain the sample.

Transfer the sample from the pipet or graduated cylinder into an appropriate container used to process the subsample further.

If the cylinder or pipet used to subsample is to be reused for other samples, thoroughly decontaminate the transfer glassware.

E.5.2.2.2 For aqueous liquids that are to be analyzed for VOA analysis, a closed system autosampler or the following may be used:

Allow the VOA vials to equilibrate to room temperature.

Suspended particulates in volatile organic samples should be allowed to settle and are not subsampled.

A gastight syringe may be inserted through the septum of the vial to withdraw the sample. Or recommend the following be done when the sample size taken is greater than 5 mL.

Remove the plunger from an appropriately sized syringe and attach a closed syringe valve. If lower detection limits are required, use a 25-mL syringe. Open the sample bottle, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL or 25.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until the analyst has determined that the first sample has been analyzed properly. If a second analysis is needed, it should be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

When sample dilution is necessary, samples can be diluted before purging. This can be performed directly in the 5-mL syringe that has been filled with reagent water through the use of appropriate microliter syringes, or with volumetric glassware, as appropriate.

Add appropriate volumes (i.e., 10.0 L) of surrogate standard solution, matrix spiking solution, and internal standard spiking solution through the valve bore of the syringe; then close the valve.

Attach the syringe-syringe valve assembly to the valve on the purging device. Open the syringe valves and inject the sample into the purging chamber. Follow appropriate procedures for purging and analysis.

E.5.2.2.3 For aqueous liquids that are to be analyzed for extractable organic analysis:

For samples destined for extractable organic analysis, it is recommended to utilize the entire contents of the 1-L sample container, and rinse the bottle with the appropriate solvent to avoid the loss of any compounds that may adhere to the walls of the container or cap.

Allow the sample and container to equilibrate to room temperature.

Mark the volume on the outside of the 1 L-sample bottle.

Note: If a bottle larger than 1 L is received, an aliquot should be poured into a graduated cylinder and then transferred into the extraction vessel. The appropriate organic solvent is then used to rinse the graduated cylinder. (Note this procedure runs the risk of generating an unrepresentative subsample.)

Make sure sample lid is attached securely and shake the sample up and down (or end over end) a minimum of three times.

Pour the sample smoothly into the extraction vessel.

Rinse the sample bottle three times with small volumes of the appropriate organic solvent and transfer these rinsates into the extraction vessel containing the sample.

Fill the sample bottle to the mark with tap water and pour the water into a graduated cylinder to determine the sample volume.

For liquid samples with suspended matter (1.0 percent), this subsampling procedure may induce analytical problems (i.e., the formation of an emulsion, or sediments clogging up separatory funnel). Deviations to these procedures (i.e., decanting the liquid with no mixing, no solvent rinsing of the bottle, etc.) should be identified within the case narrative.

E.5.2.3 Multiphase matrices - liquids/solids/sludges (both aqueous and nonaqueous). This section addresses samples considered multiphasic based on physical characteristics (mixture of solids and liquids). The choice of the procedure for handling multiphasic samples is highly dependent on project needs. Therefore, proper communication between project personnel and the laboratory is required in order to select the best approach to follow. If no clear guidance on project-specific needs is provided, analyst judgment must be used to decide what portions will be subsampled and the information documented within the case narrative. There are no specific procedures describing how samples with certain volume distributions or characteristics of liquid/solid are to be handled. However, the following general guidelines and three approaches are provided.

Nonaqueous liquids require mixing if minor particle matter is present.

Due to the different viscosities, densities, and coating properties, the weight of the subsample should typically be determined, rather than attempting to express the subsample in terms of volume. Anytime it is difficult to volumetrically measure a sample because it adheres to glassware walls, weight must be used.

If a specific and accurate weight needs to be aliquoted, a serological pipet is a good option for subsampling. Aspirate the sample, and then carefully transfer the sample into a tared vessel, controlling the addition by finger pressure on the top of the serological pipet.

E.5.2.3.1 Subsampling of samples analyzed as a single mixed phase (as received):

This approach will not provide information on the abundance of analytes in the individual phases.

The sample is mixed sufficiently to create a homogeneous sample. This is usually assessed on a visual basis. A single analysis will then be performed.

The manner in which the sample is mixed is highly dependent on sample consistency and how easily the phases mix. Some samples can simply be shaken, while others will require a spatula or mixing rod, or laboratory blender. If this is necessary, the device used to mix the sample must be noncontaminating, inert, and easily decontaminated. Usually a glass or Teflon-coated device is appropriate.

The sample is then poured, subsampled with a scoop, or transferred by some other physical means into a tared vessel and the weight of the sample is recorded. This transfer is dependent upon sample viscosity/consistency. Another consideration is not to allow the sample to resegregate into the phases when aliquoting the sample.

E.5.2.3.2 Subsampling of samples analyzed as separate phases. When the phases of a multiphasic sample are to be tested individually, the phases are separated by physical means (i.e., filtration (either pressurized or nonpressurized), centrifugation, settling, or use of a separatory funnel). The technique used is dependent on items such as laboratory capabilities, sample characteristics, and analytes of interest.

If an aliquot (subsample) of a sample is to be phase-separated, this aliquot must be representative of the original sample. This means that the solid and liquid ratio of the aliquot needs to be the same as the original sample.

To accomplish this, follow the procedures in Section E.5.2.3.1 for subsampling of samples analyzed as a single mixed phase, except that the final step is to aliquot the subsample into the device used to accomplish phase separation.

Items to consider when performing the phase separation include the following: device is noncontaminating, is nonabsorbent for analytes of interest, and does not cause the loss of analytes via other means.

Once the phases are separated, the solid and liquid ratios are determined and recorded. The two portions are transferred into either sample preparation vessels, testing vessels, or appropriate storage containers for later analysis.

If the liquid portion requires subsampling, follow the procedures listed in Section E.5.2.2. If the solid portion requires subsampling, follow procedures in Section E.5.2.1.

When phases are separated and analyzed separately, the final concentration for the total sample must be calculated mathematically. The final analyte concentration is expressed as g/mL or g/g.

E.5.2.3.3 If samples are to be analyzed as separated phases and only select phases are desired, then separate the phases as described in Section 3.5.2.3.2, discard those phases that are not of interest, and transfer portions of the phase(s) to be analyzed into appropriate preparation vessels following procedures described in Sections E.5.2.1 or E.5.2.2 as necessary.

E.5.3 Potential problems.

E.5.3.1 The most significant potential problem is the high probability of a subsample taken from a heterogenous waste being nonrepresentative.

E.5.3.2 Care must be exercised so that the introduction of contamination or potential for cross-contamination from equipment used to manipulate the sample is minimized. Full decontamination protocols must be performed on equipment between each use, and/or sufficient equipment must be available for individual sample usage. Recommend implementing the appropriate quality controls to ensure that samples are not contaminated or cross-contaminated.

E.5.3.3 Subsampling the lower phases of a multiphase liquid may pose special problems. A pipet or syringe needle passing through the lighter layers may pick up and transfer contaminants that can bias analytical results. The pipet tip or syringe needle should be wiped clean before transferring lower phase subsample to a preparation flask. Removal of the lighter layer(s) prior to subsampling may be required to obtain a representative aliquot.

E.5.3.4 Clay soil samples may be difficult to subsample with a coring-type device. Some hand coring samplers are equipped with clear plastic liner tubes that make extracting the subsample from the corer much easier. However, the goal is to obtain a representative sample. In these situations, professional judgment is required and a clean stainless steel spatula may be the tool of choice.

E.6 Decontamination Procedures

E.6.1 Scope and application. This section provides instruction on deciding on an appropriate decontamination scheme(s) for the project field sampling equipment to prevent or reduce cross-contamination of project samples. The applicability of each step in a decontamination protocol and the procedures used will depend upon the contaminants present onsite, the subsequent analysis to be performed, and the composition and type of sampling devices being decontaminated. The appropriateness of the decontamination protocol is vital to the eventual validity of the analytical results and decisions made based upon those results. All sampling equipment that contacts potentially contaminated media must be cleaned before the subsequent use of that device. Devices may include bailers, pumps, shovels, scoops, split spoons, tube samplers, augers, etc. Another approach to minimizing the potential for cross-contamination may be to dedicate or use disposable sampling equipment.

E.6.2 Decontamination procedures. Refer to Table E-3 for various stepwise decontamination protocols for sampling equipment that comes in direct contact with the sample. Each protocol begins with the detergent wash, followed by a series of chemical and water rinses, and concludes with an air-drying step. Additional guidance and protocols for the staging or setup of decontamination procedures may be found in National Institute for Occupational Safety and Health (1985) and ASTM Standards D 5088 and D 5608. To

Table E-3
Recommended Decontamination Procedures[1]

	Detergent Wash	Tap Water	Inorganic Desorbing Agents	Tap Water	Organic Desorbing Agents	Deionized Water	Air Dry
VOA							
Low MW CMPDS[2]					Methanol		
BNA/PEST/PCBS							
High MW CMPDS[2]					Hexane		
Organic Bases[3]			(1%)		Isopropyl alcohol		
Organic Acids[4]					Isopropyl alcohol		
Trace metals			(10%)				
Salts							
Acidic CMPDS							
Basic CMPDS (caustic)			(1%)				

[1] Solvent rinses vary in polarity which leads to varying solubilizing properties. The selection of appropriate solvent rinses should first consider if a known or suspected contaminant requires removal from sampling equipment. Optimum solvents for contaminants are noted. Secondly, identify whether the subsequent analytical protocol would be impacted by the proposed solvent or an impurity thereof (e.g., residual acetone present in isopropyl alcohol would be measured with certain volatile organics analysis).
[2] MW CMPDS = molecular weight compounds.
[3] Organic bases include amines, hydrazines.
[4] Organic acids include phonols, thiols, nitro and sulfonic compounds.

evaluate and document the effectiveness of the decontamination protocol, recommend the acquisition of final rinsates or wipe samples after equipment decontamination procedures are completed. Refer to Instruction G-2, Appendix G, and Instruction C-7, Appendix C, for information on the acquisition of rinsates and wipe samples, respectively.

E.6.2.1 Reagents. Reagents necessary will vary based on the protocol chosen. The following outlines general guidance for the typical reagents used to support decontamination procedures. The detergent wash is a nonphosphate detergent solution used with brushing or circulating techniques to remove gross contamination, and/or as a mild neutralizing agent. Tap water from a water system of known chemical composition is considered a control rinse water. Inorganic desorbing agents are dilute nitric or hydrochloric acid rinses. Due to their corrosive nature, the inorganic desorbing rinses may be omitted from decontamination procedures associated with metallic or stainless steel sampling devices at the discretion of project personnel. Solvent rinses (i.e., isopropyl alcohol, methanol, or hexane) are used as an organic desorbing agent. The solvent chosen must be effective in removing the organic contamination present, but must also be compatible with the subsequent analyses performed. Care should be taken to use an appropriate grade of solvent to minimize the potential introduction of impurities present in the organic desorbing rinse that may interfere or contribute to the subsequent analysis. For this reason, recommend that all solvent rinses used be appropriate grade, such as pesticide or purge-and-trap grade quality. Finally, the deionized water is organic-free reagent water.

E.6.2.2 Procedure clarifications/exceptions.

E.6.2.2.1 Table E-3 refers to the general recommended procedures used to decontaminate sampling equipment. Depending upon site contaminants, degree of contamination, analytical protocols, and composition and type of sampling equipment used, the project chemist may determine to modify or eliminate various steps of the decontamination procedures outlined in Table E-3.

E.6.2.2.2 As noted previously, the detergent wash is used in conjunction with scrubbing for gross contamination removal, followed by the appropriate rinses. For cleaning of pumping equipment or devices with inaccessible internal mechanisms, suggest circulating/flushing the system with the applicable solutions in the following order. For sampling probes used for soil gas sampling procedures, decontaminate by removing visible soil and drawing ambient air through them. Alternatively, volatiles may be baked off the soil gas probe using a portable heater. Water and solvent rinses should not be used on soil gas sampling probes. Solvent rinses for water pumping equipment should be limited to a 10 percent dilution (volume/volume) of acetone or isopropyl alcohol in water. Tubing used with peristaltic pumps may be dedicated or may be flushed with hexane, followed by a distilled water rinse depending on contaminants noted onsite. All sampling equipment should be allowed to dry prior to the next use. For this reason it is important to have sufficient sampling devices onsite so that they may be alternated. This practice will allow a thorough drying of equipment without increasing sampling downtime. If sampling equipment is not used immediately, wrap within an inert material (i.e., aluminum foil) to avoid contact with potentially contaminating materials. Equipment that does not directly contact the sample, such as large drilling equipment, drill-rig components, power augers, etc. should be cleaned with a portable power washer or a steam-cleaning machine. Finally, depending upon the project, it may be appropriate to contain spent decontamination fluids and arrange for eventual disposal as investigation-derived wastes. These containers may also be used for the eventual disposition of the materials, and therefore must comply with any potentially applicable DOT regulations.

E.6.3 Sample contaminant sources and other potential problems.

E.6.3.1 Carryover and leaching. Contaminant carryover between samples and/or from leaching of the sampling device is very complex and requires special attention. Decisions concerning the appropriateness of the material composition of the device must account for these carryover or leaching potentials, and whether these contaminants are of concern on the project. Materials potentially encountered on projects and their associated common contaminants are listed in Table E-4.

E.6.3.2 Adsorption. Contaminant adsorption is another problem that must be considered when deciding on an applicable sampling device or the appropriate composition material. This phenomenon is more critical when sampling an aqueous or gaseous media, due to the capability of lower levels of contaminant detection and the fact that the fluid matrix is more susceptible to potential contaminant transfer. PVC and other plastics are known to sorb organics and to leach plasticizers and phthalate esters. Polypropylene and other thermoplastics have been shown to sorb organics and environmental mercury efficiently and should, therefore, be avoided in sampling devices, especially tubing. For these reasons, PTFE is commonly chosen over the PVC and plastics when working with organic or mercury contaminants. In addition, some pesticides and halogenated compounds preferentially adsorb to glass surfaces. For this reason, it is recommended that when aqueous samples are taken, the sample container NOT be rinsed prior to sample collection, and the same container be rinsed with the extraction solvent after the sample has been quantitatively transferred to an extraction apparatus. Inorganics (metals) adsorption to containers is dependent upon the specific metal element, the concentration, pH, contact time, complexing agents present, and container composition. This is believed to be nominal, and proper preservation of samples should prevent this. In selecting appropriate tubing to be used for aqueous sample acquisitions, it is important to decide applicable material composition and diameter based upon the contaminant and the purpose of the data. Adsorption is less likely to occur when there is a increase in tubing diameter.

Table E-4
Materials Potentially Encountered on Projects

Material	Commonly Related Contaminants
Glass	Silicon Boron
Rigid PVC (threaded joints)	Chloroform Vinyl chloride
Rigid PVC (cemented joints)	Methyl ethyl ketone Toluene Acetone Methylene chloride Benzene Tetrahydrofuran Ethyl acetate Cyclohexanone Vinyl chloride
PVC plastic tubing	Phthalate esters Vinyl chloride Low level (zinc, iron, antimony, and copper)
Soldered pipes	Lead Tin
Stainless steel	Chromium Iron Nickel Molybdenum
Brass	Copper Zinc Tin

Appendix F
Sample Documentation and Shipment Instructions

F.1 Documentation

F.1.1 Scope and application. This section describes procedures for maintaining sample control through proper sample documentation. When samples are collected for chemical or physical characteristics analysis, documentation such as sample labels, daily contractor quality control reports (QCR), chain-of-custody and sample analysis request forms, custody seals, and field logbooks need to be completed. The information presented in this section enables maintenance of sample integrity from time of sample collection through transportation and storage. It is this documentation that will verify that samples were handled properly.

F.1.2 Documentation. The following discussion outlines standard practices and procedures to be used when documenting a sampling episode. All project-specific documentation requirements must be presented in the sampling and analysis plan (SAP). This includes identification of procedures required for field documentation, sample labeling, and the maintenance of chain-of-custody. Applicable requirements are identified in the following sections. Proper completion of all documentation with indelible ink is necessary to support the use of these records in any potential enforcement actions that may result. Protocols for corrections to documentation should not obliterate data entries, but place a single line through incorrect entry, noting corrected information, recorder's initials, and date correction was performed. Maintaining sample integrity through proper documentation is essential. Following site activities, all project documentation becomes a part of the final evidence file. These records should be maintained for a certain period of retention time. The documentation retention time requirements of a project must be presented within the SAP and may be based on the use of the data, funding source, or regulatory authority.

F.1.2.1 Daily contractor quality control reports (QCR). During the field investigation or remedial action activities, daily contractor QCRs should be prepared daily, dated, signed by the project contractor quality control representative, and sent to USACE at a rate specified in the scope of work or specifications. With respect to geotechnical and chemical procedures, these reports should include weather information at the time of sampling, field instrument measurements, calibrations, identification of all field and control samples taken, departures from the approved SAP necessary, deviations from approved geotechnical procedures (such as well installation or drilling), any problems encountered, and instructions from Government personnel. Any deviations that may affect data quality objectives must be conveyed to U.S. Army Corps of Engineers (USACE) personnel (technical manager, project geologist, project chemist, etc.) immediately. The following should be attached to the daily contractor QCRs: quality assurance (QA) sample tables that match up primary, replicate (quality control (QC)/QA), and other field control samples (e.g., blanks), copies of chain-of-custody forms, field-generated analytical results, and any other project forms that are generated. Additional documentation requirements of the daily contractor QCRs are outlined in Engineer Regulation (ER) 415-1-302 and Corps of Engineers Guide Specification (CEGS) 01451.

F.1.2.2 Field logbooks. Sampling situations vary widely. No general rules can specify the exact information that must be entered in a field logbook for a particular site. However, the logbook should contain sufficient information to enable the sampling activity to be reconstructed without relying on the collector's memory. Project field logbooks should be bound and have numbered, water-resistant pages. Record the site name and project name and number on inside front cover of logbook. All pertinent information regarding the site and sampling procedures must be documented as near to real-time as possible. At the conclusion of each day, the person maintaining the logbook should sign and date the day's

documentation entries. Notations should be made in logbook fashion, noting the time and date of all entries. Information recorded in other project documents (e.g., boring logs, well installation/development logs, or drum logs) should not be repeated in the field logbook, except in summary form to avoid transcription errors. Logbooks should be kept in the field team member's possession or in a secure place during field work. Following site activities or if the logbook is completely filled, the logbook becomes a part of the project final evidence file as noted previously. The technical planning team may also elect to establish documentation requirements that follow a more uniform organization than a field logbook. These documentation requirements would include the use of project forms. This approach helps enhance consistency in the information recorded and streamlines the documenting process. Any forms proposed for use should be task specific and should incorporate appropriate topics from those identified as follows. All forms must be presented in the project SAP. The following are some suggested topics to include in the field logbook:

- Name and exact location of site of investigation or interest.
- Name and title of person maintaining logbook (author).
- Date and time of arrival and departure at site location.
- Purpose of site visit or sampling activity.
- Name and address of field contact. This may also include information on access agreements.
- Names and responsibilities of all persons on site.
- Names, affiliations, and purpose of all site visitors.
- Level of personal protective equipment worn at the site.
- Weather conditions on the day of sampling, and any additional environmental conditions or observations pertinent to field activities.
- Field instrumentation or equipment used, and purpose of use (i.e., health and safety screening, sample selection for laboratory analysis). Note source, quality, or lot numbers for any supplies or reagents (e.g., sample containers, preservatives, reagents, water for field blanks/field control samples, and decontamination procedures). Retain any certificates or information supplied with the equipment used.
- Type of waste, suspected waste concentrations if known, and sample matrices to be handled.
- Document the sample collection method and any sample handling procedures such as filtration, compositing, and executed preservation techniques used.
- Document the sample location. If a compositing scheme is used, clearly identify appropriate locations for all sample aliquots included within each composite sample. Prepare a dimensional sketch of the general surroundings of the sampling area (site), and/or support with other forms of documentation (i.e., photographic log). Sample identification numbers should correspond directly with sample locations.

- Identify sample numbers, volumes, and containers (number, size, type) used for each sample collected. Note the date and time of each sample, identify any associated QC samples, or any factors that may affect the quality.

- Record any field measurements, field screening/analytical results generated, calibration methods used, field results, and QC information.

- Identify decontamination procedures employed for sampling equipment.

- Document appropriate references to maps and photographic logs of the sampling site.

- Record information on scheduling modifications, change orders, sampling or drilling decisions/changes.

- Describe the number of shipping coolers packed, note chain-of-custody (COC) numbers or attach a copy of COC, and record the mode of transportation and applicable tracking numbers.

- Record name and address of all receiving laboratories.

- Maintain appropriate documentation for investigation-derived wastes. Note contents and volumes of waste generated, storage, and disposal methods used.

F.1.2.3 Documenting sampling points and locations. The exact locations of sampling points should be documented for purposes of generating an accurate representation of the site conditions using the data generated to date, defining data gaps, and identifying potential future data needs. A monument should be chosen at each site to act as a stationary reference point from which all sampling points can be measured using a compass and measuring tape. If a building or other stationary structure exists, its comer may act as this reference point. If no monument exists, it will be necessary to create one. A piece of wood, approximately 5 cm by 5 cm (2 in. by 2 in.), should be hammered into the ground to almost ground level, making it difficult to remove and thus assuring its permanence. The stake should then be marked with flagging tape or fluorescent paint. When applicable, sampling points associated with coordinates that are referenced to a position on the earth must comply with ER 1110-1-8156. ER 1110-1-8156 requires geospatial data to be documented using the Federal Geographic Data Committee's content standards for digital geospatial metadata. Geospatial data are nontactical data, referenced either directly or indirectly to a location and boundaries on the earth. Additional guidance on geospatial data systems may be found in EM 1110-1-2909. To establish a sampling point, the following procedure is recommended:

- Standing at the monument, facing the sampling point, use the compass hairlines to determine the degree of direction.

- Ensure that the line of sight runs from the monument, through both hairline needles on the compass, to the sampling point.

- When first establishing the sampling point, record the degree and direction reading from the compass in the field logbook, along with the distance measurement from the monument to the sampling point.

F.1.2.4 Photographic documentation. All sampling points should be documented on film. A film record of a sampling event allows positive identification of the sampling point. In some cases, a photograph

of the actual sample collected may also be required. Photographs are the most accurate and convenient record of field personnel observations. Photographs taken to document sampling points should include two or more reference points to facilitate relocating the point at a later date. Keeping a record of photographs taken is crucial to their validity as a representation of an existing situation. Photographic documentation is invaluable if the sampling and subsequent analytical data end in litigation, enforcement, or cost recovery actions. In addition to photographs, video coverage of a sampling episode can be equally as valuable as or even more valuable than photographs because it can be used to prove that samples were taken properly as well as verify the location at which they were taken. Video coverage can be used as a record of site conditions and can give those who have not been onsite an idea of the circumstances. For each photograph taken, the following items should be noted in the field logbook:

- Date.
- Time.
- Photographer (signature).
- Name of site.
- General direction faced and description of the subject.
- Sequential number of the photograph and the roll number.
- Site photo map (see Figure F-1).

F.1.2.5 Sample documentation

F.1.2.5.1 Sample labels. Sample labels are required for properly identifying samples and evidence. All samples must be properly labeled with the label affixed to the container prior to transportation to the laboratory. It is also recommended that samples be photographed so that labels are clearly readable for later identification. Information on sample labels should include, but not be limited to, the following:

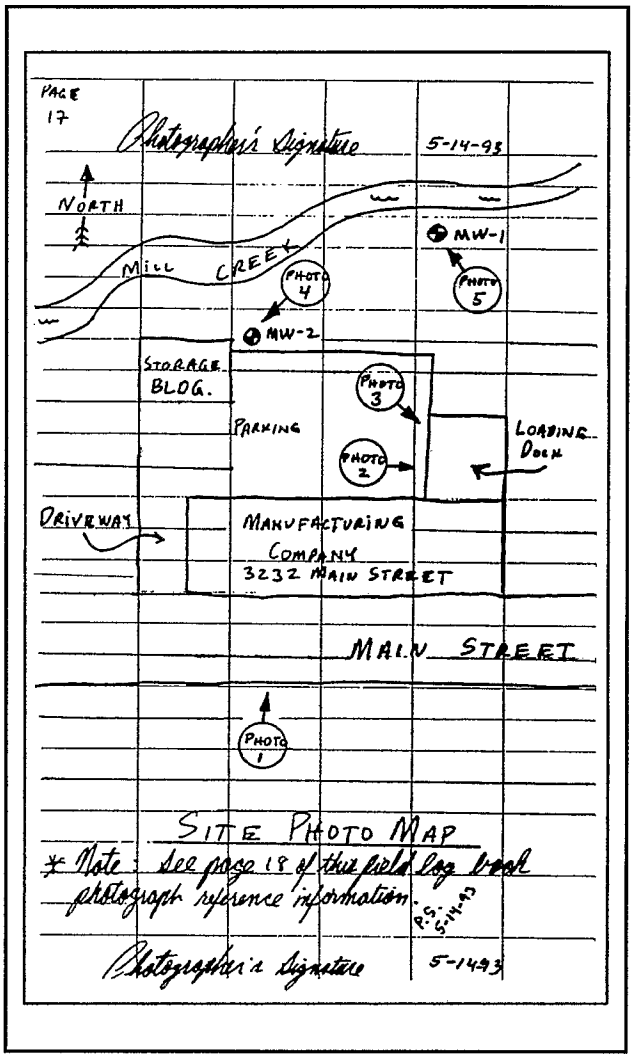

Figure F-1. Site photo map

Project Code. An assigned contractor, project number, site name.

Station Number. A unique identifier assigned to a sampling point by the sampling team.

Sample Identification Number. Each sample, including field control samples, collected for a project should be assigned a unique number. This assigned number incorporates information on the sample type and date as noted in Section F.1.2.5.2.

Samplers. Each sampler's name and signature or initials.

Preservative. Whether a preservative is used and the type of preservative.

Analysis. The type of analysis requested.

Date/Time. Identify the date and time the sample was taken.

Type of Sample. The type of sample should be identified as discrete or composite.

F.1.2.5.2 Sample numbering. A sample numbering system should be used to identify each sample collected and submitted for analysis. The purpose of the numbering system is to assist in the tracking of samples and to facilitate retrieval of analytical results. The sample identification numbers for each sampling effort should be used on sample labels, sample tracking matrix forms, COC forms, field logbooks, and all other applicable documentation. A listing of all sample identification numbers should be recorded in the field logbook. The sampling numbering system may vary depending upon the number and type of samples that will be collected at the site. An example of a sample numbering system follows. Location and sample identification numbers should consist of the following designations to identify the location (AABBB-CC), sample sequence number, date (MMDDYY), and sample depth interval for soils (00-00):

- For soil: AABBB-CC/MMDDYY/00-00
- For water: AABBB-CC/MMDDYY
- For QC samples: AABBB-CC/MMDDYY

Example: SB001-01/081492/08-10 = Soil Boring SB001 Sample Number 1, sampled on August 14, 1992, from a sample depth interval of 8 to 10 ft (2.4 to 3 m). Duplicate samples should be numbered in sequential order. For example, a duplicate sample collected from this soil boring example would have a designation as follows: SB001-02/081492/08-10. Each sample collected must be assigned a unique sample number. Sample numbers should change when the media or location changes. Sample numbers should not change because different analyses are requested. For example, water samples collected at the same location, date, and time for volatile organics, semivolatile organics, and metals analyses would all have the same sample number, although the various sample aliquots would be collected in different containers.

F.1.2.5.3 Chain-of-custody (COC). COC procedures provide documentation of the handling of each sample from the time it is collected until it is destroyed. COC procedures are implemented so that a record of sample collection, transfer of samples between personnel, sample shipping, and receipt by the laboratory that will analyze the sample is maintained. Records concerning the cleaning of empty sample containers, container shipment from the laboratory to the site, and security of empty containers at the site should also be maintained. The COC record (Figure F-2) serves as a legal record of possession of the sample. The COC record is initiated with the acquisition of the sample. The COC record remains with the sample at all times and bears the name of the person (field investigator) assuming responsibility for the samples. The field investigator is tasked with ensuring secure and appropriate handling of the bottles and samples. To simplify the COC record and eliminate potential litigation problems, as few people as possible should handle the sample or physical evidence during the investigation. A sample is considered to be under custody if one or more of the following criteria are met:

CHAIN-OF-CUSTODY RECORD

PROJ. NO.	PROJECT NAME					NO. OF CON-TAINERS	REMARKS
SAMPLERS: (Signature)							
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION		

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	

Figure F-2. Chain-of-custody form

The sample is in the sampler's possession.

The sample is in the sampler's view after being in possession.

The sample was in the sampler's possession and then was locked up to prevent tampering.

The sample is in a designated secure area.

In addition to the COC record, there is also a COC (custody) seal. The COC seal (Figure F-3) is an adhesive seal placed in areas such that if a sealed container is opened, the seal would be broken. The COC seal ensures that no sample tampering occurred between the field and the laboratory analysis.

F.1.2.5.4 Transfer of custody and shipment. All sample sets should be accompanied by a COC record. When transferring possession of samples, the individual receiving the samples should sign, date, and note the time that he/she received the samples on the COC record. This COC record documents transfer of custody of samples from the field investigator to another person, other laboratories, or other organizational units. Samples must be properly packaged for shipment and delivered or shipped to the designated laboratory

for analyses. Shipping containers must be secured by using nylon strapping tape and custody seals (Instruction F-2). The custody seals must be placed on the container so that it cannot be opened without breaking the seals. The seal must be signed and dated by the field investigator. When samples are split with a facility, state regulatory agency, or other government agency, the agency representative must sign the COC record, if present. All samples should be accompanied by the COC record. The USACE tracking number (e.g., laboratory information management system (LIMS) number) that is used in conjunction with the Government QA sample shipment must be written on the COC record of the QA sample. The original and one copy of the record will be placed in a plastic bag taped to the inside lid of the secured shipping container. One copy of the record will be retained by the field investigator or project leader. The original record will be transmitted to the field investigator or project leader after samples are accepted by the laboratory. This copy will become a part of the project file. If sent by mail, the package should be registered with return receipt requested. If sent by common carrier, an air bill should be used. Receipts from post offices and air bills should be retained as part of the documentation of the COC. The air bill number or registered mail serial number should be recorded in the remarks section of the COC record.

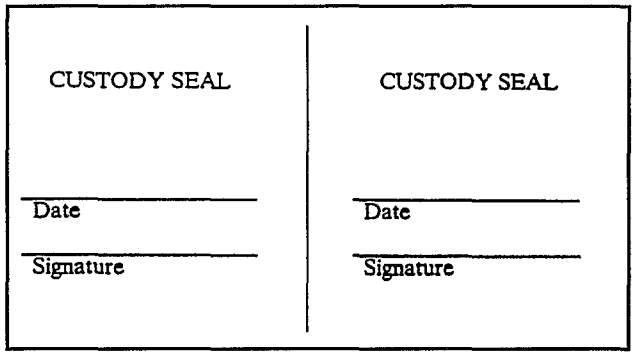

Figure F-3. Chain-of-custody seal

F.1.2.5.5 Sample analysis request. To ensure that proper analysis is performed on the samples, additional paperwork may need to be filled out, as required by the laboratory performing the analysis. This form identifies samples by number, location, and time collected and allows the collector to indicate the desired analysis. This form should act as a supplement/confirmation to the COC record and laboratory contacts made prior to the sample event initiation.

F.1.3 QA/QC requirements.

F.1.3.1 Corrections to documentation. All original data recorded in field logbooks and on sample labels, COC records, and receipt-for-samples forms are written in waterproof ink. If an error is made on an accountable document, corrections should be made simply by crossing out the error and entering the correct information. The erroneous information should not be obliterated. Any error discovered on a document should be corrected by the person who made the entry. All corrections must be initialed and dated.

F.1.3.2 Photographs. The photographer should review the photographs or slides when they return from developing and compare them with the photographic log to confirm that the log and photographs match.

F.1.4 Potential problems. Although most sample labels are made with water-resistant paper and are filled out using waterproof ink, inclement weather and general field conditions can affect the legibility of sample labels. It is recommended that after sample labels are filled out and affixed to the sample container, the label should be covered with wide clear tape. This will preserve the label and keep it from becoming illegible. In addition to label protection, COC and analysis request forms should be protected when samples are shipped in iced coolers. Typically, these forms should be placed inside a ziplock bag or similar waterproof protection and taped to the inside lid of the secured shipping container with the samples.

F. 2 Packaging and Shipping Procedures

F.2.1 Scope and application. This section describes procedures for properly packaging and shipping environmental and hazardous waste samples, as well as the shipment of preservatives used in the environmental sampling. Guidelines for proper container and preservative selection can be found in Appendix B. Personnel that are involved in packaging, shipping, and receipt of samples must be aware of Department of Transportation (DOT) regulations, know when to apply them, and know what procedures are needed to support this application. Personnel who ship samples considered a DOT hazardous material (HM) must be trained in accordance with the requirements set in 49 CFR 172.704. The following procedures identify packaging and shipping requirements for environmental and hazardous waste samples that are both DOT-regulated and nonregulated. Further information is presented for shipping carriers (i.e., FedEx, UPS) that use the International Air Transportation Association (IATA) regulations to govern domestic and international shipments. Shipping procedures for common preservatives and decontamination fluids are also addressed. Finally, it should be noted that DOT regulations also apply to the shipment of asbestos samples.

F.2.2 Procedures for shipping environmental samples. Environmental samples are defined as those samples collected from environmental matrices such as soil, groundwater, or sediments. The following sections identify packaging and shipping requirements for environmental samples that are unpreserved or preserved by acid/base/chemical addition. The following general procedures apply to the packaging of all environmental samples:

- Verify that the sample label is complete and adequately identifies the items described in Instruction F-1.

- Verify that each sample cap/lid is secured on the bottle, and place each sample in a plastic bag. For multiple volatile organic analysis (VOA) vials, all vials from each sample location should be placed in a small plastic bag at a minimum. Evidence tape or custody seals may be placed over the sample lid and container, or over the seal of the bag for additional security, if desired.

- Squeeze as much air as possible from the bag, and seal the bag. Trip blanks are packaged in the same manner as that for aqueous VOA samples.

- Prepare the shipping container for use. For a commercial cooler, this includes taping the drain plug shut inside and out, and lining the cooler with a large plastic garbage bag. Place approximately 7.5 cm (3 in.) of inert packing material in the bottom of the liner. Place vermiculite or perlite on the bottom if the materials are liquid. Alternative shipping containers may be used if approved by project technical personnel.

- Place the samples upright in the lined cooler or storage container in such a way that the samples will not touch each other during shipment. Add inert packing material as necessary to ensure separation of samples.

- With the exception of aqueous metals analyses, all environmental samples should be shipped to the laboratory on ice and chilled to 4 °C ± 2 °C. If any of the shipped samples require cooling, place double bags of ice around the containers. Also include a 40-mL VOA vial filled with water for use as a temperature blank for the laboratory.

- Fill the cooler with packing material and tape the inner liner shut. NOTE: Do not use "environmentally friendly" peanuts made of starch to pack containers of liquids. These packing materials will dissolve when they get wet or moist.

- Place the paperwork (Instruction F-1) being sent to the laboratory inside a plastic bag and tape it to the inside of the cooler lid. Include a copy of the COC form in the paperwork sent to the laboratory. The sampler keeps one copy of the COC form. Include any additional paperwork to notify the laboratory of project information (laboratory notification checklist), or if a sample is suspected of containing any substance for which laboratory personnel should take safety precautions.

- Close the cooler and seal it with strapping tape.

- Place at least two custody seals on the outside of the cooler (one on the front and one on the back). More custody seals may be used at the discretion of the sampler.

- Prepare standard air bill paperwork for shipment of the samples to the laboratory. Personnel should be aware of carrier weight or other policy restrictions.

F.2.2.1 Unpreserved environmental samples. Environmental samples that are shipped unpreserved are not considered a hazardous material by DOT if they do not exhibit a DOT hazard class. This exempts them from DOT regulation during transportation to the laboratory. In general, this applies to soil/sediment and aqueous samples preserved by cooling. Follow the general procedures identified in Section F.2.2 for packaging and shipment of unpreserved environmental samples.

F.2.2.2 Preserved environmental samples. All samples that are preserved by the addition of chemicals are subjected to greater scrutiny when defining whether DOT regulations apply and the appropriate packaging and shipping requirements necessary. Care should be exercised when adding any chemicals to environmental samples for preservation purposes. Samples must be observed, noting any chemical reactions that take place, and following with any contingency measures needed to obtain a representative sample. Most importantly, add only the amount of preservative needed to achieve the required preservation criteria. Excessive preservation may make an otherwise unregulated sample subject to DOT hazardous materials regulations. On average, testing of water samples arriving at commercial laboratories has shown that between 30 and 50 percent of the samples are excessively preserved and may have been improperly packaged and shipped. Samples in this category may also be considered a Resource Conservation and Recovery Act (RCRA) characteristic or listed waste. However, an exclusion from manifesting and hazardous waste marking requirements exists under RCRA for shipping environmental samples to the laboratory. The RCRA exclusion can be found in 40 CFR 261.4(d). Materials that are Toxic Substances Control Act (TSCA) regulated (polychlorinated biphenyls (PCBs)) may be shipped under exclusions found in 40 CFR 761.65(I). However, these exemptions do not pertain to DOT regulations, and compliance with DOT regulation is still required. Field personnel should also realize that even though the DOT may allow for these items to be shipped by air, other international regulations governing air carriers (such as IATA/International Civil Aviation Organization (ICAO)) may further limit or forbid materials allowed by DOT. The shipper must also comply with all packaging regulations. These regulations will be found in 49 CFR 171.101 and 49 CFR 173. Packaging and shipping requirements for preserved environmental samples are determined based on whether the preserved samples exhibit the same physical or chemical characteristics that initially defined the preservative as a DOT hazardous material. Follow the guidance presented in Section F.2.2.2.1 and in Sections F.2.2 or F.2.3 to determine the appropriate shipping and packaging requirements to apply.

F.2.2.2.1 Excepted preserved environmental samples. The DOT definition of a corrosive material is based on the destruction of intact animal skin. The DOT packing group (PG) associated with these samples determines the packaging and shipping procedures necessary, which are based on the time frame of observed tissue damage. Alternative testing to the animal testing has been developed and is accepted by DOT to support an application for exception from DOT classification and regulation. In the spring of 1997, USACE performed alternative testing to support the exception from the determination of acid/base preserved samples as a DOT corrosive classification. The testing showed that samples preserved in the manner described in Table F-1 and packaged as outlined in paragraph F.2.2 did not meet the definition of corrosive materials as prescribed by DOT under 49 CFR 173.136 (a)(1), 173.137 (a,b,c(1)). Based on this information, environmental samples meeting these criteria may be shipped as non-DOT regulated material following procedures outlined in Section F.2.2 under the USACE number DOT-E10904. Further information on this exception should be obtained from the USACE Hazardous, Toxic, Radioactive Waste - Center of Expertise (HTRW-CX) prior to its application or use. For consistency, the proper preparation of sample preservatives is outlined as follows for some common preservatives used in conjunction with environmental sampling:

Acid preservatives:

(1) Hydrochloric acid — 500 mL water + 2.0 mL 12N hydrochloric acid

(2) Nitric acid — 500 mL water + 2.0 mL 15.7N nitric acid

(3) Sulfuric acid — 1000 mL water + 1.0 mL 18M sulfuric acid

- Base preservatives:

Sodium hydroxide — 500 mL water + 1.0 mL 30% sodium hydroxide

Table F-1 identifies the required pH ranges for sample preservation to meet standard U.S. Environmental Protection Agency methods, and to avoid meeting the definition for a particular DOT hazard classification and packing group. Samples preserved in the field should be verified for correct pH to assess the utilization of this exception. This may become problematic if the testing materials used (i.e., pH test strips) do not distinguish pH values to the required accuracy. Based on the range limits established, it is mandatory to use pH paper capable of a minimum of 0.5 pH unit resolution, such as the use of short-range pH paper. However, due to the proximity of several of these values, it is recommended a pH meter be used to allow resolution to 0.1 pH unit. Field personnel are cautioned that, based on specific acid type, if pH is adjusted below the acid range limit or above the basic range limit, preserved samples will be regulated by DOT as a hazardous material and will require proper shipping papers, marking, and labeling as identified in Section F.2.2.2.2.

Table F-1
Preserved Sample pH Ranges Needed for DOT Exemption

Preservative Used	Acceptable Sample pH Ranges for DOT Exemption Status[1]
Hydrochloric acid	pH = 1.43 to 2.00
Nitric acid	pH = 1.33 to 2.00
Sulfuric acid	pH = 1.31 to 2.00
Sodium hydroxide	pH = 12.00 to 12.58

[1] Per USACE exemption DOT-E10904

F.2.2.2.2 Acid/base preserved environmental samples. Environmental samples that have been overpreserved may meet the DOT definition of hazardous materials (i.e., corrosive liquid). When applicable, these materials must be shipped in proper DOT-approved containers, unless the shipper uses limited-quantity exceptions, or if the shipper has determined through testing that the material does not meet the definition of a DOT corrosive (Section F.2.2.2.1). DOT and IATA regulations should be reviewed to determine appropriate packaging and shipping requirements, and if a limited-quantity exemption exists. If a limited-quantity exemption does exist, the volume of hazardous material to be shipped is evaluated to determine if it exceeds this threshold limit. Refer to Table F-2 for information on the proper shipping name (PSN) and the limited quantities associated with some common types of acid preservation. When the total volumes of shipped material are less than the limited quantities noted, then the materials may be shipped as a limited quantity following the sample packaging and shipment procedures included in Section F.2.3, with "Limited Quantity" or "Ltd Qty." recorded on the shipping papers after the PSN. Table F-3 summarizes general requirements necessary to ship DOT-limited quantities. If the shipper does not meet these requirements, then the samples are considered fully regulated DOT hazardous material, and all DOT requirements as defined in 49 CFR 171-178 must be met.

Table F-2
PSN and Limited Quantities for Common Preservatives, Decontamination Fluids, and Potentially Hazardous Samples[1]

Chemical Compound/Sample/ Material	PSN	Lmt Qty[2]	Lmt Qty[3]
Sulfuric acid >51%	Sulfuric Acid, with more than 51% sulfuric acid, 8, UN1830, PGII	1 liter	30 Liters
Sulfuric acid <51%	Sulfuric Acid, with not more than 51% sulfuric acid, 8, UN2796, PGII	1 liter	30 Liters
Nitric acid >70%	Nitric Acid, with more than 70% nitric acid, 8, UN2031, PGI	Forbidden	2.5 Liters
Nitric acid <70%	Nitric Acid, with less than 70% nitric acid, 8, UN2031, PGII	Forbidden	2.5 Liters
Nitric acid solution (10%)[4]	Nitric Acid, other than red fuming with less than 20% nitric acid, 8, UN2301, PGII	1 liter	30 liters
Nitric acid solution (1%)[4]	Nitric Acid, other than red fuming with less than 20% nitric acid, 8, UN2301, PGII	1 liter	30 liters
Nitric acid preserved samples	Corrosive Liquids, Acidic, Inorganic, NOS (Nitric Acid), 8, UN3264, PGIII	5 liters	60 liters
Hydrochloric acid	Hydrochloric Acid Solution, 8, UN1789, PGII	1 liter	30 liters
Hydrochloric acid preserved samples	Corrosive Liquids, Acidic, Inorganic, NOS (Hydrochloric Acid), 8, UN3264, PG III	5 liters	60 liters
Phosphoric acid	Phosphoric Acid, 8, UN1805, PGIII	5 liters	60 liters
Sodium hydroxide solution	Sodium Hydroxide Solution, 8, UN1824, PGII	1 liter	30 liters
Sodium hydroxide preserved sample	Corrosive Liquids, Basic, Inorganic, NOS (Sodium Hydroxide), 8, UN3266, PGIII	5 liters	60 liters
Isopropyl alcohol	Isopropyl alcohol or Isopropanol, 3, UN1219, PGII	5 liters	60 liters
Methanol	Methanol, 3, UN1230, PGII	1 liter	60 liters
Hexanes	Hexanes, 3, UN1208, PGII	5 liters	60 liters
Sodium bisulfate, aqu sol	Bisulfate, aqueous solution, 8, UN2837, PGII	1 Liter	30 liters
Fuel ID/Tank Samples	Flammable Liquids, NOS (fuels), 3, UN1993, PGIII.	60 liters	220 liters

[1] Personnel should be aware that the following represents only a few of the possible Proper Shipping Names (PSNs) available for shipping hazardous materials. Not all materials field personnel may have to ship are covered by these examples. Shippers are advised to review the DOT hazardous materials table prior to shipping.
[2] Passenger Aircraft/Railcar.
[3] Cargo Aircraft Only.
[4] Nitric acid solutions at 20% acid are recognized by the ICAO and IATA, and are addressed in 49 CFR 171.11.

Table F-3
Limited Quantity Packaging and Shipping Paper Criteria

Parameter	Class 3	Class 6.1	Class 8	Class 9
Outer Package (max gross wt)	30 kg/66 lb	30 kg/66 lb	30 kg/66 lb	30 kg/66 lb
Outer Package Markings:				
1. PSN				
2. UN#				
3. Orientation Arrows - liquids				
4. Address				
5. RQ	( )	( )	( )	( )
6. Other Markings: Marine Pollutant, Cargo Air Craft, etc.	( )	( )	( )	( )
Label	Air: Flammable Liquid Other: excepted	Air: Poisonous Other: excepted	Air: Corrosive Other: excepted	Air: Class 9 Other: excepted
Inner Package Size Limit:				(Not PG specific)
1. PGI	0.5 L	N/A	N/A	4.0 L/5.0 kg
2. PGII	1.0 L	N/A	1.0 L/1.0 kg	
3. PGIII	5.0 L	4.0 L/5.0 kg	4.0 L/5.0 kg	
Shipping Paper Entries:				
1. Gross wt				
2. Number of packages				
3. Proper shipping description				
4. "Ltd Qty"				
5. Marine Pollutant	( )	( )	( )	( )
6. RQ	( )	( )	( )	( )

Note:
Entry Required
( ) Required if definition criteria are met

F.2.2.2.3 Methanol preserved samples. Soil and sediment samples may require preservation with methanol if samples will be tested for volatile organics by the high-level method. In this case methanol is added neat to the sample and is, therefore, considered a hazardous material. DOT and IATA regulations should be reviewed to determine appropriate packaging and shipping requirements, and to determine if a limited-quantity exemption exists. If so, the volume of hazardous material to be shipped is evaluated to determine if it exceeds this threshold limit. Refer to Table F-2 for information on the PSN, and limited quantities associated with methanol. Additional information on limited quantities should be referenced from IATA regulations when applicable. Again, refer to Table F-3 for a summary of general requirements necessary to ship DOT-limited quantities. For instance if the total volume of methanol included within sample jars packaged in the shipping container does not exceed 1 L, then one may ship under the exception found in 49 CFR 173.150 (b)(2) for limited quantities. This is supported by the DOT Hazardous Materials Table, which identifies the methanol quantity limitation for passenger aircraft or railcar as 1 L (0.3 gal). When the total volumes of shipped material are less than the limited quantities noted, then the materials may be shipped as a limited quantity. "Limited Quantity" or "Ltd Qty." must be recorded on the shipping papers after the PSN if the exception is used. Sample packaging and shipment should follow procedures included in Section F.2.3. When greater than 1 L is included, sample packaging and shipment procedures noted within 49 CFR 172.701, Hazardous Materials Table, must be followed. Recommend packaging shipping containers to maintain the volume of methanol below the thresholds noted in Table F-2. Project personnel are also

encouraged to check with the shipping carrier used, to verify that additional, more stringent policy requirements do not exist for the shipment of flammable liquids such as methanol. Another option available under DOT is the small-quantity exception found in 49 CFR 173.4. Specifically, 49 CFR 173.4(a)(1)(i) states the maximum quantity of material per inner container is limited to 30 mL for authorized liquids, other than Division 6.1, Packing Group I materials (i.e., poisons). In other words, for Method 5035 (EPA/SW-846) preserved samples, if one has less than or equal to 30 mL of methanol or bisulfate aqueous solutions (sodium bisulfate) per inner (sample) container, this material is not subject to any other requirements of the hazardous materials regulations except those in 49 CFR 173.4. DOT hazard classes covered by this exception include Class 3, Division 4.1, Division 4.2 (PGII and PGIII), Division 4.3 (PGII and PGIII), Division 5.1, Division 5.2, Division 6.1, Class 7, Class 8, and Class 9. In addition to the 30-m container limit, additional restrictions and requirements apply. Personnel taking this exception should review 49 CFR 173.4 carefully. Finally, no 49 CFR 173.21 (forbidden) materials may be packaged, the gross weight of the completed package cannot exceed 29 kg (64 lb), and the package cannot be opened or altered until it is no longer in commerce (transport). The shipper must certify conformance with the referenced sections by marking the outside of the package with the statement "*This package conforms to 49 CFR 173.4*" or alternatively until 1 October 2001 with the statement "*This package conforms to the conditions and limitations specified in 49 CFR 173.4.*" Further, the shipper must indicate on the air waybill under nature and quantity of goods, "*Dangerous goods in Excepted Quantities.*" The IATA also requires the application of an "Excepted Quantities" label. This label contains the certification language previously identified. Label entries include shipper signature, title, date, address, and indication of the hazard class and associated United Nations (UN) number.

F.2.2.2.4 Quantity limitations. One final restriction to note is that while 49 CFR 173.4 does not have a total net quantity limitation, IATA Dangerous Goods Regulations (DGR Section 2.7.5.2) does. For packing group II materials (i.e., methanol or sodium bisulfate) the total net quantity limit is 500 mL. This equates to 33 inner (sample) containers (i.e., VOA vials) containing up to 15 m of preservative per outer package (cooler). When shipping DOT hazardous materials by air, shippers have additional restrictions that are identified in Columns 9A/9B of the 49 CFR 172.101 Hazardous Materials Table. Net quantity limits of methanol for passenger and cargo aircraft are 1 L and 60 L, respectively. The net quantity limits for sodium bisulfate solutions are 1 L and 30 L, respectively. Shippers should note that *these* quantities exceed the IATA small-quantity exception. Therefore, if the volume of preservative (methanol or sodium bisulfate solution) is kept less than 30 mL per inner (sample) container *and* total net quantity per outer package (cooler) is limited to 500 mL, then quantity limits given in DOT Hazardous Materials Regulations or IATA Dangerous Goods Regulations are not an issue provided packaging conforms with 49 CFR 173.4.

F.2.2.2.5 HTRW-CX assistance. The HTRW-CX has coordinated with the Logistics Support Activity Packaging, Storage, and Containerization Center at Tobbyhanna Army Depot, Tobbyhanna, PA, to develop a standard 49 CFR 173.4 tested and certified packaging. Materials needed to assemble these sample and shipping packages are readily available to field personnel from local hardware or retail stores. The protocol established is available for USACE personnel use by contacting HTRW-CX for additional information.

F.2.3 Procedures for shipping hazardous samples.

F.2.3.1 Hazardous samples are defined as those that are typically highly contaminated, such as oils, sludges, discarded products, and items that exhibit a hazard as defined by DOT, or if it is suspected that they may be explosive, reactive, poisonous, toxic, flammable, or corrosive. Samples with visual evidence of explosives content (e.g., TNT flakes) should be considered suspect and managed appropriately. Hazardous waste samples taken for chemical analyses are normally taken in small volumes with preservation limited to cooling. Packaging and shipping requirements for hazardous samples are typically determined based on

any known contaminants or characteristics of the samples. In several cases, field screening techniques may be used to identify the packaging requirements necessary. The shipment of these samples to the laboratory may also be considered exempt from regulation under RCRA/TSCA as previously described and as referenced from 40 CFR 261.4(d) and 40 CFR 761.65(I). However, these exemptions do not pertain to DOT or IATA regulations. DOT-defined hazardous material samples must be packaged and shipped in accordance with all applicable DOT regulations, including those establishing sample container types and specifications, marking, labeling, placarding requirements, and the preparation of associated shipping papers. In most cases, the shipper will be able to package and ship these samples under the limited-quantity requirements found in column 8A, Packaging Exceptions, of the Hazardous Materials Table (49 CFR 172.101). If no limited-quantity exceptions are found in column 8A, then field personnel should use the following guidelines to determine appropriate packaging and shipping requirements.

F.2.3.2 Initially, the shipper must determine the appropriate DOT hazard class. If the shipper is unable to determine the proper DOT hazard class, due to the unknown nature of the sample, the shipper must consult 49 CFR 171-177 to determine the proper hazard class and shipping name. The next step is to look at column 8A or 8B to determine the proper outer and (if required) inner nonbulk packaging requirements and any applicable exceptions. As it is safest to assume that the materials to be shipped do meet the definitions of a DOT hazard class, the outer container must be properly labeled and marked and the shipper must comply with all regulations concerning shipping papers and placarding. The cooler, or other outer package if considered an overpack (49 CFR 171.8 and 49 CFR 173.25), must be marked and labeled accordingly. For air shipment of samples that meet the definition of a DOT-hazardous material, the shipper must also use the quantity limitations found in column 9 of the Hazardous Materials Table (49 CFR 172.101). Further recommend that the transportation by air be designated as cargo aircraft. By specifying cargo aircraft, the shipper is permitted to ship larger volumes of material in a single outer container with less stringent regulatory requirements. The requirements for packaging, packing, and shipping for hazardous samples are outlined as follows. (Note: The following protocol should be used for the shipment of hazardous samples only if the shipper is taking a limited-quantity exception, unless the "paint can" is a UN specification container (i.e., 1A2, 1B2, etc). If the shipper is not taking a limited-quantity exception, UN performance-oriented packing requirements apply. Do not assume paint cans are UN specification packages unless they are marked in accordance with 49 CFR 178.503.)

- Ensure sample container label is complete, and adequately identifies the items prescribed in Instruction F-1.

- Verify each sample cap/lid is secured onto the bottle. Tape shut the lid onto sample containers, and place each sample in a plastic bag. Activated carbon may also be placed with the sample within the plastic bag to prevent cross-contamination.

- Place evidence tape or custody seals over the sample lid and container, or over the seal of the bag for additional security, if desired.

- Squeeze as much air as possible from the bag, and seal the bag.

- Place each bottle upright in a separate paint can. Fill the paint can with vermiculite, and affix the lid to the can. The lid must be sealed with metal clips or with filament or evidence tape; if clips are used, the manufacturer typically recommends six clips.

- Place DOT Orientation arrows on the can to indicate which end is up.

- Mark each with the proper DOT shipping name and identification number for the sample. These can be found referenced in the Hazardous Materials Table. The information may be placed on stickers or printed legibly. A liquid sample of an uncertain/unknown nature is shipped as a flammable liquid with the shipping name "FLAMMABLE LIQUID, N.O.S." and the identification number "UN1993." A solid sample of uncertain nature is shipped as a flammable solid with the shipping name "FLAMMABLE SOLID, N.O.S." and the identification number "UN1325." If the nature of the sample is known, 49 CFR 171-177 is consulted to determine the proper marking, labeling and packaging requirements. Always use DOT-approved outer containers to ship samples that meet or are suspected to meet the definitions of a hazardous material.

- Place the cans upright in a cooler that has had its drain plug taped shut inside and out, and has been lined with a garbage bag. Place vermiculite or perlite on the bottom if the materials are liquid.

- All hazardous samples should be shipped to the laboratory on ice and chilled to 4 °C ± 2 °C.

- Place additional inert packing material (styrofoam peanuts) in the cooler to partially cover the sample bottles. If samples are required to be shipped to the laboratory with ice, place bags of ice around the containers. The cooler must then be filled with packing material and the inner liner taped shut. NOTE: Do NOT use "environmentally friendly" peanuts made of starch to pack containers of liquids. These packing materials will dissolve when they get wet or moist.

- Place the paperwork going to the laboratory inside a plastic bag and tape it to the inside of the cooler lid. A copy of the COC form should be included in the paperwork sent to the laboratory. The sampler keeps one copy of the COC form. The laboratory should be notified if a sample is suspected of containing any substance for which laboratory personnel should take safety precautions.

- Close the cooler and seal with strapping tape. Place at least two custody seals on the outside of the cooler (one on the front and one on the back). More custody seals may be used at the discretion of the sampler.

- Place the following markings on the top of the cooler:

  (1) Proper shipping name (49 CFR 172.301).

  (2) DOT UN/North America identification number (49 CFR 172.301).

  (3) Shipper/consignee's name and address (49 CFR 172.301).

- Place the following labels on top of the cooler (49 CFR 172.406(e)):

  (1) Appropriate hazard class label (adjacent to PSN).

  (2) "Cargo Aircraft Only" (as needed, per 49 CFR 172.101).

  (3) Certification statement: "Inside (inner) packages comply with the prescribed specifications"

- Place orientation markings on two opposite vertical sides indicating "This Way Up" in addition to the markings and labels described in preceding item (49 CFR 172.312).

- Use restricted-article air bills for shipment. The "Shipper Certification for Restricted Articles" section is filled out as follows:

  (1) Number of packages or number of coolers

  (2) Proper shipping name

  (3) Classification

  (4) Identification number

  (5) Net quantity per package or per cooler

  (6) Radioactive materials section (leave blank)

  (7) Note passenger or cargo aircraft

  (8) Name and title of shipper (printed)

  (9) Emergency telephone contact number within 24 to 48 hr

  (10) Shipper's signature

IATA Dangerous Goods Regulations list the hazard classes for many compounds. If the materials to be shipped cannot be found on the list, it may be necessary to use a more generic (not otherwise specified (NOS)) description. IATA regulations apply mainly to international shipment of hazardous materials by air. However a number of overnight domestic carriers (such as FedEx and UPS) also use IATA regulations to govern domestic shipments. Quantity limitations concerning hazardous materials shipments are usually the same as DOT; however, exceptions exist. IATA regulations must be reviewed if a domestic carrier requires IATA quantity limitations. Examples of PSNs that may be appropriate for samples are included in Table F-2.

F.2.4 Procedures for shipping preservatives and decontamination fluids. Preservatives and decontamination solvents used in environmental and hazardous waste sampling are often hazardous materials. These materials must also be stored and shipped in accordance with all applicable regulations. The preferred method for transporting preservatives to the site, many of which are DOT hazardous materials, is to order them from a chemical supply company and have that company ship the materials directly to the sampling site or base of operations. This reduces the liability and regulatory compliance issues that must be dealt with for field personnel. If project personnel must ship the materials defined as DOT-hazardous for use as preservatives or decontamination fluids, compliance with all applicable DOT regulations, including proper shipping containers, container markings, placarding, packaging, labeling, and shipping paper requirements is required. When Government personnel will transport preservatives and decontamination fluids to the field in a Government vehicle via highway only, the less stringent 49 CFR 173.6, Materials of Trade Exceptions, may apply. Further information on the exception can be obtained from the HTRW-CX. Procedures for shipping preservatives and decontamination fluids are outlined as follows:

- Determine the proper DOT shipping name (PSN) for the materials to be shipped. The PSN for hazardous materials will be found in 49 CFR 172.101, Hazardous Materials Table. The PSNs for the most common preservatives are given in Table F-2.

- Determine whether there is an exception for limited quantities for the material(s) to be shipped. Refer to column 8A in 49 CFR 172.101, Hazardous Materials Table, under the PSN for the specific material that needs to be shipped.

- If there is a limited-quantity exception and the materials to be shipped meet those quantity limitations for the transportation methods shown in column 9A/9B of the Hazardous Materials Table and comply with the proper packaging requirements as shown in column 8A, then the materials may be shipped as a limited quantity. "Limited Quantity" or "Ltd Qty." must be recorded on the shipping paper after the PSN if the exception is used. Some of the quantity limitations of the more common chemicals used as preservatives in environmental samples are given in Table F-2.

- If the material does not have an exception as a limited quantity, the next step is to determine the requirements to properly package the material under DOT regulations. Refer to 49 CFR 172.101, Column 8B. In this section (packaging authorizations for nonbulk packaging), a three-digit (***) number is present. To find the proper section for packaging authorizations, see 49 CFR 173.(***). For example, under Sulfuric Acid, the three-digit number found in column 8B is 202. So the shipper should look under 49 CFR 173.202 for the packaging authorizations for shipping sulfuric acid. The materials to be shipped MUST comply with the required packaging.

- Determine whether there are placarding requirements for the shipment. Refer to 49 CFR 172.500 for this information.

- Ship the materials following the criteria established.

F.2.5 Potential problems.

F.2.5.1 Field personnel should be aware that there are discrepancies for nitric acid in the shipping name tables for DOT and ICAO/IATA. IATA/ICAO allow the shipment of $\leq$20 percent nitric acid via passenger aircraft and allow these concentrations to be shipped as a limited quantity. DOT does not acknowledge this PSN entry, but does acknowledge ICAO technical instructions. Refer to 49 CFR 171.11 for additional details.

F.2.5.2 Note that excessive sample preservation is very likely to bring an environmental sample into the DOT hazardous materials regulatory realm. Depending on the specific inorganic acid used as a preservative, a difference of 0.5 pH unit (e.g., pH 1.0 versus pH 1.5) will likely trigger all DOT hazardous materials communication standards and regulations. The ranges noted in Table F-2 are provided to help field personnel make the appropriate decisions associated with the classification of preserved environmental samples for transportation.

F.2.5.3 Individual samples known to contain or are highly suspected of containing PCBs at greater than or equal to 0.45 kg (1 lb) or greater than or equal to 1 percent by weight are regulated in the air mode. In this instance DOT hazardous materials regulations are applicable. Further, PCBs are regulated as a marine pollutant, which requires additional notations on the shipping paper. Readers are referred to 49 CFR 172.203.