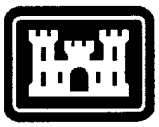
CEMP-RA Engineer Manual 200-1-3	Department of the Army U.S. Army Corps of Engineers Washington, DC 20314-1000	E200-1-3 1 February 2001
	Engineering and Design REQUIREMENTS FOR THE PREPARATION OF SAMPLING AND ANALYSIS PLANS	
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US Army Corps
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EM 200-1-3
1 February 2001

ENVIRONMENTAL QUALITY

Requirements for the Preparation of Sampling and Analysis Plans

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ENGINEER MANUAL

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DEPARTMENT OF THE ARMY
U.S. Army Corps of Engineers
Washington, DC 20314-1000

EM 200-1-3

CEMP-RA

Manual
No. 200-1-3

1 February 2001

Engineering and Design
REQUIREMENTS FOR THE PREPARATION OF SAMPLING AND ANALYSIS PLAN

1. Purpose. This manual provides guidance for the preparation of project-specific sampling and analysis plans (SAP) for the collection of environmental data. In addition, default sampling and analytical protocols are included which may be used verbatim or modified based upon project-specific data quality objectives (DQOs). The goal of this manual is to promote consistency in the generation and execution of sampling and analysis plans and thus to help generate chemical data of known quality for its intended purpose.

2. Applicability. This manual applies to all USACE Commands having responsibility for sampling and analysis of environmental samples. This includes, but is not limited to, USACE activities pursuant to and in support of execution of the following programs or sponsors: Defense Environmental Restoration Programs; Base Realignment and Closure; Superfund; Civil Works, Military Construction, installation environmental compliance; Defense Logistics Agency; Department of Energy; work for others; and any construction projects involving hazardous, toxic, and radioactive waste (HTRW).

3. Distribution Statement. This manual is approved for public release; distribution is unlimited.

FOR THE COMMANDER:



ROBERT L. DAVIS
Coldnel, Corps of Engineers
Chief of Staff

IO Appendices
(See Table of Contents)

This manual supersedes EM 200-1-3, dated 1 September 1994.

DEPARTMENT OF THE ARMY
U.S. Army Corps of Engineers
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EM 200-1-3

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Engineering and Design
REQUIREMENTS FOR THE PREPARATION OF SAMPLING AND ANALYSIS PLANS

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Chapter 1
Introduction

1.1 Purpose

This manual provides guidance for the preparation of project-specific sampling and analysis plans (SAP) for the collection of environmental data. In addition, default sampling and analytical protocols are included which may be used verbatim or modified based upon project-specific data quality objectives (DQOs). The goal of this manual is to promote consistency in the generation and execution of sampling and analysis plans and thus to help generate chemical data of known quality for its intended purpose.

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This manual applies to all USACE Commands having responsibility for sampling and analysis of environmental samples. This includes, but is not limited to, USACE activities pursuant to and in support of execution of the following programs or sponsors: Defense Environmental Restoration Programs; Base Realignment and Closure; Superfund; Civil Works, Military Construction, installation environmental compliance; Defense Logistics Agency; Department of Energy; work for others; and any construction projects involving hazardous, toxic, and radioactive waste (HTRW).

1.3 References

Required and related publications are listed in Appendix A.

1.4 Explanation of Acronyms and Terms

Acronyms and special terms used in this manual are explained in the glossary.

1.5 Functional Equivalencies

1.5.1 The SAP has replaced the document that was formerly known as the Chemical Data Acquisition Plan. SAPs prepared in accordance with the guidance provided by this manual are intended to be functionally equivalent to U.S. Environmental Protection Agency (USEPA) sampling and analysis plans, field sampling plans, and quality assurance project plans prepared under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and to data collection quality assurance plans and data management plans prepared under the Resource Conservation and Recovery Act (RCRA).

1.5.2 The SAP is divided into two parts: a field sampling plan (FSP) and a quality assurance project plan (QAPP). The FSP addresses the field activities, including all aspects of sampling, drilling, monitoring well installation, and any field data gathering activities. The QAPP addresses the data quality objectives, analytical methodologies, specific quality assurance (QA) and quality control (QC) activities, laboratory requirements, and data assessment activities designed to achieve the data quality goals of the project.

1.5.3 This manual contains requirements for format and contents of the SAP and instructions for specifying and executing sampling, analysis, and related tasks for measurement of chemicals in the environment. Certain situations may require that the SAP be written differently from the format described in this manual. For example, work performed on behalf of certain USEPA regions may follow

a different SAP format from that prescribed within this manual. Many states have their own regulations regarding underground storage tanks, which may also impact SAP preparation. This manual complements existing USACE guidance as referenced in Appendix A.

1.6 Discussion

1.6.1 The SAP is a document prepared by an architect-engineer (A-E) firm, a remedial action contractor, or USACE to describe the project requirements for all field and laboratory activities, any data assessment activities, and contract deliverables related to the reporting of chemical data for HTRW remedial activities. When the SAP is prepared under contract (e.g., with an A-E firm), it is done in response to a scope of work (SOW) prepared by USACE that describes specific tasks and objectives of the project. Investigative projects include preliminary assessment/site inspections (PA/SI), remedial investigation/feasibility studies (RI/FS), engineering evaluation/cost analyses, RCRA facility assessments, RCRA facility investigations, and corrective measure studies.

1.6.2 In addition to investigative projects, this manual may be used for developing plans for data collection activities such as predesign bench and/or pilot studies, remedial action or post-closure monitoring, perimeter or offsite ambient air monitoring, etc. The size and complexity of a project will be reflected in the SAP. Although the guidance in this manual is applicable to radioactive wastes, unexploded ordnance, chemical warfare agents, and biological wastes, additional guidance may be necessary to prepare SAPs involving these materials. When chemical data are acquired, the SAP is one component of the overall project work plan. SAPs are required for each contractor work order. All details of field and laboratory activities must be described in the FSP and QAPP, respectively. These documents must be submitted to the appropriate USACE technical staff for review, comment, and approval. Once approved, the SAP represents the standard to which all activities are compared to assure compliance.

1.7 Relationship of SAP to the Project Work Plan

Per other USACE guidance on scoping HTRW investigative projects involving generation of analytical data, the SAP is included as an attachment to the project work plan. For those projects in which a work plan is not required, such as certain remedial actions, the SAP must be a stand-alone document.

1.7.1 Project work plan. The project work plan is an umbrella document that addresses, but is not necessarily limited to, the following subjects.

1.7.1.1 Project background. This section includes a brief summary of the site: size and location; ownership history; authority under which the work is to be performed; and the purpose and scope of the work plan. The inclusion of maps noting the location of the project within a state or county is recommended.

1.7.1.2 Site description and history. This section includes a description of the geology of the site, building structures, if any, topography of the site, etc. Other relevant information may include annual precipitation, prevailing wind direction, and site hydrology. It also includes a brief history of the site in terms of former activities, reported spills, and waste disposal practices that may have contributed to potential contamination over the years.

1.7.1.3 Previous investigations. This section includes discussion of previous investigation activities and other response activities at the site and also any problems and/or data anomalies.

1.7.1.4 Project objectives (long- and short-term). This section explains the purpose of the project: the regulatory framework under which the work is being conducted, what goals are to be met; and what questions are to be answered. In the case of a PA/SI, the objectives might include a determination of whether there is enough evidence to support the need for an RI/FS. In the case of an RI/FS, the objectives might include site contamination characterization in terms of extent and concentration, risk assessment, and the screening of remedial action alternatives. Applicable or relevant and appropriate requirements should be addressed. Much of the information in this chapter is helpful in guiding the preparation of the SAP.

1.7.1.5 Data gaps. This paragraph provides information regarding data gaps that need to be filled in order to make project decisions, such as defining the extent of contamination and choosing remedial action alternatives.

1.7.1.6 Data quality objectives (DQOs). This paragraph describes how data will be used to make project decisions. This paragraph may serve as a general scoping guide for data acquisition activities defined in the SAP.

1.7.2 SAP. The attachments to the project work plan (SAP, site safety and health plan, etc.) provide details of the specific data collection activities that are designed to support the objectives of the project, as set forth in the work plan. Information in the project work plan and SAP should not be redundant. Project-specific DQOs, including measurement quality objectives (MQOs) for precision, bias, representativeness, completeness, comparability, and sensitivity are addressed in the SAP. MQOs are applicable to both sampling and analytical portions of the project.

1.8 Technical Project Planning

1.8.1 As prescribed within Engineer Manual (EM) 200-1-2, Technical Project Planning Process, USACE and/or contractor technical planning teams are responsible for developing project-specific data collection programs that define the quality and quantity of data needed to perform all the engineering and scientific evaluations required for the project.

1.8.2 Initially, USACE and/or the contractor must identify the appropriate data users needed for the project. Data users involved are project dependent, and may include the customer, regulators, risk assessor, compliance or regulatory specialist, remedial design engineers, an attorney, etc. Data users will determine initial data needs in order to perform specific evaluations and make the engineering and scientific judgments required to complete the necessary activities leading to site closeout.

1.8.3 Sampling and analysis data implementors provide input to planning specific data collection tasks and are responsible for task execution based upon the project data needs. Data implementors are also chosen based upon the project and include technical personnel such as a geologist, hydrogeologist, chemist, statistician, sampling personnel, etc. This manual provides guidance to these data implementors for preparing SAPs for conducting field and analytical work and is a source of standard operating procedures (SOPs).

1.8.4 This manual provides both data users and implementors with a vehicle to prescribe sampling and analytical protocols necessary to achieve data quality objectives dictated by the technical project planning process.

1.9 Overview of Manual

1.9.1 This manual consists of four chapters, ten appendices and a glossary. Chapter 2 presents guidelines for use of the manual. Chapter 3 discusses format and content requirements of FSP and QAPP components of SAP. Chapter 4 lists guidelines for developing sampling and analysis protocols when those protocols in Appendices C, D, E, F, G, H, and I are not appropriate. Appendix B presents a table of holding times, preservatives, and sample containers for various parameters. Appendix C presents instructions for collecting environmental samples from various media. Appendix D gives hazardous waste sampling instructions. Appendix E gives sample handling instructions. Appendix F presents sample documentation and shipping instructions. Appendix G describes field QA/QC elements and procedures. Appendix H presents guidance on the application of field analytical technologies. Appendix I discusses general and method-specific chemical analysis requirements. Appendix J provides a review checklist for SAPs.

1.9.2 This manual will be revised as needed by modifying/adding instructions to incorporate changes and innovations within the environmental community, as well as changes in USACE policy.

Chapter 2
Utilization of this Engineer Manual

2.1 General

This chapter discusses how this manual may be used to prepare, review, and implement an SAP. It also describes how the manual may be used by USACE personnel as a source for specifying sampling instructions when preparing the SOW or, in the case of a site-remediation project, the plans and specifications for the project. How to execute an SAP and verify compliance with the field and analytical procedures specified in the SAP are briefly described also.

2.2 Scope of Work Preparation

2.2.1 This engineer manual contains information that may be used during the technical planning of projects and generation of project SAPs. It is a USACE mission to characterize and remediate HTRW-contaminated sites in an efficient, cost-effective, and technically sound manner. To attain this goal, technical planning teams should utilize other USACE guidance for standard outlines on scoping HTRW investigations, chemical quality assurance, and HTRW technical project planning. Refer to EM 200-1-6, Chemical Quality Assurance for HTRW Projects; and EM 200-1-2, Technical Project Planning (TPP) Process, for information on scoping, the QA elements available for Chemical Data Quality Management (CDQM) execution, and technical project planning protocols, respectively. With the assistance of these guidance documents, the technical team must at a minimum generate a project SOW that clearly identifies project goals, associated data needs, and application of QA elements based upon the project goals designed to reach site closeout. The team may decide to further clarify the effort within the SOW by identifying specific requirements for implementation of defined data collection options or a specific data collection program, or appropriate performance and/or measurement quality objectives for QC samples and corrective actions necessary.

2.2.2 The appendices of this manual contain sampling and analytical SOPs that may be considered when identifying the data collection options and/or program. These include, but are not limited to, various matrices sampling and sample handling techniques, analytical methods, field and laboratory QA/QC protocols, documentation requirements, and appropriate references. DQO statements that describe the data collection design for sampling and analysis of each matrix must be defined.

2.2.3 USACE personnel may specify in the SOW or plans and specifications the individual instructions or SOPs that should be used in the SAP, or they may simply reference this manual as a source of SOPs. Contractors providing services for USACE may have their own sampling and analytical SOPs that would be suitable for a given project. In these cases, this manual provides a format for structuring the contractor's instructions for inclusion in the SAP. This will ensure continuity in the HTRW program. If project-specific objectives and strategies cannot be satisfied by any of the instructions in the relevant appendices, references for alternate sampling and analytical methods are included in Appendix A. Paragraph 4.4 of this manual discusses how to develop new sampling and analysis instructions.

2.3 SAP Preparation

The following three-step approach is suggested to prepare the SAP. The SOW or plans and specifications will specify the extent to which the architect/engineer or remedial action contractor will interact with USACE during the three-step approach.

2.3.1 Step 1: Consult with technical planners. Contractors working under agreement with USACE should initially consult with USACE technical planners to obtain project information. This step is not applicable to USACE in-house projects because USACE technical planners (technical managers and/or project scientists/engineers) actually prepare the SAP. USACE technical planners may interact directly with their customer to obtain information. However, contractors working under an agreement with USACE should consult with USACE technical planners to obtain important facility information, data from previous investigations, and information regarding site constraints.

2.3.2 Step 2: Review appropriate project documentation/literature. Before writing the SAP, perform a thorough review of all appropriate project documents. Foremost is the SOW or plans and specifications for the current work effort. These documents contain results from the technical project planning process as outlined in EM 200-1-2. As noted previously, the level of specificity outlined within these SOWs may vary from outlining general project goals with appropriate references to specifying sampling and analytical requirements to meet the project-defined data quality objectives for each matrix. Other applicable references required for background information should be identified within the SOW also. These may include, but are not limited to, applicable engineer regulations and guidance documents, regulatory program and status reports from previous studies and investigations, construction data, ownership/operational histories, site maps and photographs, information on regional and site geology, hydrogeology, hydrology, topography, ecology, climatology, demographics, and current and future land use.

2.3.3 Step 3: Review requirements for format and contents of SAPs. Chapter 3 discusses the general format and content requirements for the FSP and QAPP portions of the SAP. A good working knowledge of these requirements is necessary to understand the type of information required to draft an SAP and determine if additional sources of information are required. If it is determined that the sampling and/or analytical methods in the appendices of this manual or other existing references are not appropriate, Chapter 4 of this manual can be used to develop site-specific protocols.

2.4 SAP Review/Approval/Distribution

2.4.1 Review. The SAP should be reviewed to determine whether it will provide data that satisfy customer and technical planner data needs, whether it satisfies the data use and data quality objectives, and whether it is compatible with all site constraints. Reviewers should use the "review checklist" found in Appendix J as a guide for reviewing the SAP. This checklist is a very general guide and contains information that typically should be included in an SAP. USEPA/state guidance documents for preparing CERCLA/RCRA investigative plans may also be consulted.

2.4.2 Approval. After the SAP has been reviewed, the document can be accepted as is or returned to its authors for review comment resolution. Once the SAP has been approved, appropriate personnel sign the signature page, and the SAP becomes a contractual document. The USACE personnel that will sign the SAP will be determined on a project-specific basis by the technical planning team. It is recommended that the USACE technical manager sign the title page of the SAP and that the USACE chemist sign the title page of the QAPP. Any deviations from the approved document must receive

written approval from USACE. In addition, there may be significant changes in the project that necessitate that the SAP be appended or modified. Similar procedures of review and approval for those modified sections would be necessary prior to execution of the modifications.

2.4.3 Distribution. Once approved, the final SAP or its modifications must be distributed to all parties as defined within the SOW contract. These may include USACE technical manager, primary or referee (QA) laboratory(ies), any regulatory authorities, customer, and any subcontractors (i.e., drilling or sampling firms, data validation firms, etc.).

2.5 SAP Execution and Compliance

This manual may be used by USACE contractors and USACE oversight personnel as a guide for either executing the SAP or monitoring compliance with the SAP. Before data collection activities are implemented with either contractor or USACE resources, an approved SAP must be in place. All laboratories must have an approved SAP in order to be aware of project analytical requirements, must be able to meet and perform all aspects of the required chemical analyses, and must provide data reportables as specified within the QAPP portion of the SAP. Execution of the SAP must be performed in compliance with the approved SAP. Field personnel must be adequately trained for their duties and possess a full understanding of all aspects of the SAP. Sampling personnel shall ensure that proper field equipment is available and in good condition, and sample collection and handling procedures, including sample preservation, are performed in accordance with the prescribed sampling instructions or SOPs. A liaison between the field and laboratory (however named) shall be identified and shall ensure smooth transition of all samples from the field to the laboratory. Liaison duties may include implementation of proper sample packaging and shipping procedures and any communication or notification with the laboratory. Safety and health requirements and practices as defined in ER 385-1-92, Safety and Occupational Health Document Requirements for Hazardous, Toxic and Radioactive Waste (HTRW) and Ordnance and Explosive Waste (OEW) Activities, must be adhered to throughout all phases of environmental sampling operations. During the execution of the SAP, compliance is monitored by USACE by conducting field, desk, and laboratory audits. In addition, implementation of the project-defined QA elements (i.e., field control samples, referee laboratory analyses, data assessment procedures, etc.) allows additional insight into sampling and analysis activities. While data collection activities are being performed, the sampling team should communicate daily with appropriate USACE personnel regarding project status by submitting appropriate documentation as outlined in the SOW. Lastly, the final report review provides an opportunity for verification of DQO attainment, data assessment, and identification of any value-added procedures or corrective actions necessary. EM 200-1-6, Chemical Quality Assurance for HTRW Projects, provides guidance on field and laboratory techniques for assessing chemical data, identification of any limitations on data use, and recommended documentation procedures. The use of statistics during the data assessment may also be recommended by the regulatory authority.

2.5.1 Quality assurance (QA) elements. As defined in EM 200-1-6, there are several QA elements that may be applied to an HTRW project to ensure proper execution of CDQM. These include, but are not limited to, validation of chemistry laboratories, proper technical review/approval of project documents (i.e., SAP), field and laboratory audits, QA sample handling verification, referee lab (QA) sample analysis, use of single- and double-blind performance evaluation (PE) samples, data review and/or data validation, magnetic tape audits, and generation of Chemical Quality Assurance Reports and Chemical Data Quality Assessment Reports. The project SOW or, in the case of a site remediation project, the plans and specifications must define the appropriate QA elements to be applied to the project, the frequency of application, and any notification, contingency, or corrective action protocols necessary

in the event of deficiency or failure. This information must then be reiterated within the project documents to clearly define QA implementation procedures.

2.5.2 Audits. USACE personnel should conduct field and desk audits for all field sampling activities conducted as part of the HTRW program. Laboratory audits may be performed in conjunction with the laboratory validation process; district personnel are also encouraged to perform precontract or preaward system audits of the laboratory to ensure proper communication and awareness of project DQOs are in place. Combining these audits to increase overall effectiveness of the audit is recommended. The audits of field activities should be performed whether the project is executed in-house or by contractors for any phase of work from initial investigation to postclosure monitoring. This oversight is necessary to ensure that approved procedures, as specified in the SAP, are used to perform the work. Field audits include monitoring critical activities, such as well installation and well development, placement of other types of sample access devices (e.g., passive soil gas collection media), decontamination of equipment used to generate samples or other activities that could cause cross-contamination, sample collection from all media (i.e., air, ground water, surface water, soil, sediment, and waste), and postsample collection activities (packaging/shipping). Field audits should be scheduled as early in the activity as possible to identify procedures that could cause problems with the sampling and analytical results. Checklists included within EM 200-1-6 may be used to enhance consistency and completeness of the field audits conducted; as well as providing an aid for documenting the audit results. Another mechanism for monitoring field activities as they occur is to perform desk audits. This is usually done by reviewing daily contractor QC reports, chain of custodies, and field logs while the field activities are in progress. The SOW or plans and specifications should have a requirement stating that these reports be supplied on a periodic basis (e.g., daily or weekly).

2.5.3 Corrective action. The SAP should also address notification and corrective actions that should be followed by field and laboratory personnel if there are deviations from the SAP or problems with samples upon receipt at the laboratory. Typical problems/deviations include, but are not limited to, the following: improperly preserved samples, improper chain-of-custody documentation, broken sample containers, sample relocation, insufficient volume, etc. As a minimum requirement, the SAP should state that significant changes to or deviations from the approved SAP should not be made without the written approval of USACE. The QAPP should also describe corrective action procedures that are required if field and/or analytical procedures are found to deviate from the requirements in the SAP. Example corrective action measures include, but are not limited to, resampling with additional analysis of new samples, reanalysis of existing field or QC samples, or proper data qualification. Appendix I provides additional guidance on corrective action requirements of the laboratory.

Chapter 3
Sampling and Analysis Plan - Format and Contents

3.1 General

This chapter contains general guidance for the format and contents of an SAP, including a brief discussion of each of the major elements. Several additions have been incorporated in order to accommodate the technical requirements identified within the USEPA guidance EPA QA/R-5 and EPA QA/G-5. However, due to the disparity in the adoption or enforcement of this guidance among the various USEPA regions and State regulating agencies, the existing two-component format has been retained. The use and application of this or other project plan formats are at the discretion of the USACE technical team members. However, the technical contents of the plans should be equivalent.

3.2 Format Requirements

3.2.1 The SAP consists of two parts: an FSP and a QAPP. The FSP provides guidance for all fieldwork by defining in detail the sampling and field data-gathering methods to be used on the project. The QAPP describes the chemical data quality objectives, analytical methods and measurements, QA/QC protocols necessary to achieve the DQOs, and data assessment procedures for the evaluation and the identification of any data limitations. The FSP and QAPP should be submitted as a single document (although they may be bound separately to facilitate the use of the FSP in the field).

3.2.2 The FSP and QAPP are prepared prior to any field activities, but the FSP and QAPP may be amended or revised several times during the investigation activities using the protocol outlined in section 2.4 of this manual. Alternatively, the WP and associated SAP may develop a contingency-based, more flexible strategy to data gathering activities. This dynamic approach must outline a decision logic, which when supported by field analysis results promote decisions being made in the field about the subsequent site activities and/or refinement of the conceptual site model (CSM). Issues to be addressed within the SAP include establishing key field personnel experience requirements, the level of decision-making empowered to key field personnel, and communication protocols between the field and project stakeholders. For projects that encompass several subsites or involve a long-term contract (i.e., Total Environmental Restoration Contract), it may be beneficial to generate a comprehensive SAP that covers all aspects of sampling and analytical requirements conducted at a project and/or site. Then this document can be amended for individual delivery orders by generating an abbreviated, project-specific SAP. This addendum to the SAP must clearly identify the current effort's DQO, applicable matrices, site-specific sampling and analysis requirements, and any deviations from the comprehensive SAP. Information previously addressed within the comprehensive SAP may be referenced in the project-specific SAP addendums. When this approach is utilized, all SAP addendum topics referencing the comprehensive SAP must be verified by the USACE technical planning team during the document review process. Inadequate coverage of topics must be resolved prior to the SAP addendum execution. Preparatory phase inspections (field audits) must ensure that all appropriate plans (comprehensive and addendum SAPs) are available onsite and field personnel are familiar with procedures included within both.

3.2.3 Table 3-1 lists the typical elements that should appear in the FSP and QAPP. Depending upon the size and/or complexity of the project, all of the elements identified in Table 3-1 may not be appropriate for every project. In these instances, the format may be abbreviated or modified to accommodate the individual project activities.

Table 3-1
SAP Format Requirements

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Distribution List
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I Field Sampling Plan (FSP)

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1.0 Project Background
  1.1 Site History and Contaminants
  1.2 Summary of Existing Site Data
  1.3 Site-Specific Definition of Problems
2.0 Project Organization and Responsibilities
3.0 Project Scope and Objectives
  3.1 Task Description
  3.2 Applicable Regulations/Standards
  3.3 Project Schedule
4.0 Nonmeasurement Data Acquisition
5.0 Field Activities by Area of Concern (AOC)
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    5.1.2 Field Procedures
      5.1.2.1 Equipment
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      5.1.2.4 Field Progress/Interpretation Reporting
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      5.3.1.2 Sample Collection and Field and Laboratory Analysis
      5.3.1.3 Upgradient, QA/QC, and Blank Samples and Frequency
    5.3.2 Monitoring Well Installation
      5.3.2.1 Drilling Methods and Equipment
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        5.3.2.2.3 Surface Completion
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        5.3.2.3.2 Soil Sampling and Rock Coring During Drilling
        5.3.2.3.3 Geophysical Logging

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5.3.2.3.8 Cement/Bentonite Grout Placement
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5.3.2.3.10 Protective Cover Placement
5.3.2.3.11 Well Identification
5.3.2.3.12 Well Development
5.3.2.3.13 Well Survey
5.3.2.3.14 Alignment Testing
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5.3.2.4.5 Photographs
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Table 3-1 (Continued)

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- 6.3 Photographic Records
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  - 6.4.1 Sample Numbering System
  - 6.4.2 Sample Labels and/or Tags
  - 6.4.3 Chain-of-Custody Records
- 6.5 Field Analytical Records
- 6.6 Documentation Procedures/Data Management and Retention

7.0 Sample Packaging and Shipping Requirements

8.0 Investigation-Derived Wastes (IDW)

9.0 Field Assessment/Three-Phase Inspection Procedures
- 9.1 Contractor Quality Control (CQC)
- 9.2 Sampling Apparatus and Field Instrumentation Checklist

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II Quality Assurance Project Plan (QAPP)

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2.0 Data Assessment Organization and Responsibilities

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- 3.1 Data Use Background
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C List of Abbreviations and Acronyms

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Site Remedial Objectives
Previous Analytical Data Summary
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Proposed Monitoring Well Information
Sample Container Preservation and Holding Time Requirements
Names and Addresses of Owners of Property Near the Site
Sample Container Quantities
Summary of Sample Matrices and Locations
Summary of Number of Samples and Analyses

Example List of Figures

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Proposed Monitoring Well and Onsite Sample Locations
Proposed Offsite Sample Locations
Monitoring Well Construction
Investigation Schedule

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3.3 Content of Major Elements

The FSP describes the field activities to be performed and defines the procedures and methods that must be used to collect field measurements and samples. Issues include collection of geophysical data; drilling of soil borings; installation of ground water monitoring wells; and procedures for collection of multimedia samples, field control samples, and any field measurements. The FSP also addresses the sample packaging and shipping requirements, proper handling and disposal of investigation-derived wastes (IDWs), field documentation procedures, corrective action procedures, and the project schedule.

The QAPP focuses primarily on the analytical methods and QA/QC procedures that are used to analyze the samples and manage the data. The QAPP should include the organization and responsibilities of project laboratory and data assessment personnel; QA objectives; sample receipt, handling, custody, and holding time requirements; analytical procedures, equipment preventive maintenance, calibration, internal quality control procedures, and performance/system audits; data reduction, review, and reporting; and data assessment, data useability, and DQO reconciliation. The recommended requirements for the contents of FSPs and QAPPs are discussed in the following subsections. Additional information may be obtained from EPA QA/R-5, EPA QA/G-5, and other references provided in Appendix A.

3.3.1 Title page. The title page should be the first page of the SAP. The following items should appear on the title page: the name of the document, site name and location, USACE contract number (if applicable), regulatory authority under which the activities are being performed (CERCLA, RCRA, etc.), and date of preparation. If necessary, due to project modifications, the SAP may be assigned a document and/or revision number. If tasks performed under the SAP are executed by a contractor, Figure 3-1 is an example signature block that should appear at the bottom of the title page. If the recommended signatures are difficult to acquire, it is suggested that a statement be added that the approved SAP was provided to the appropriate parties (i.e., laboratories, drillers, data assessment personnel, etc.), identifying names of recipients and the date.

COMMITMENT TO IMPLEMENT THE ABOVE SAMPLING AND ANALYSIS PLAN

Contractor's Project/Task Manager (print)	Signature	Date
Contractor's QC Manager (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date

* Commitment signature is required for any ancillary sampling, analytical, or data assessment support provided by a contractor or subcontractor. For example, the Contractor's laboratory QC manager or director should sign the title page if analytical services are provided.

Figure 3-1. Example signature block

3.3.2 Distribution list. This list should include all recipients of the SAP, and any addendums or modifications thereto.

3.3.3 Table of contents. This should be a very general table of contents that outlines the layout of the SAP. Table 3-2 is an example.

3.3.4 Field sampling plan (FSP). An FSP should include the following:

Table 3-2
SAP Table of Contents Example

Introduction

I Field Sampling Plan
Title Page
Table of Contents
1.0 Project Background
2.0 Project Organization and Responsibilities
3.0 Project Scope and Objectives
4.0 Nonmeasurement Data Acquisition
5.0 Field Activities by Area of Concern (AOC)
6.0 Field Operations Documentation
7.0 Sample Packaging and Shipping Requirements
8.0 Investigation-Derived Wastes (IDW)
9.0 Field Assessment/Three Phase Inspection Procedures
10.0 Nonconformance/Corrective Actions

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1.0 Project Laboratory Organization and Responsibilities
2.0 Data Assessment Organization and Responsibilities
3.0 Data Quality Objectives
4.0 Sample Receipt, Handling, Custody, and Holding Time Requirements
5.0 Analytical Procedures
6.0 Data Reduction/Calculation of Data Quality Indicators
7.0 Laboratory Operations Documentation
8.0 Data Assessment Procedures

Appendices

A References
B Standard Forms to be Used
C List of Abbreviations and Acronyms

3.3.4.1 Title page. The FSP should have an abbreviated title page that includes the name of the document (e.g., Phase I Remedial Investigation Field Sampling Plan), document and/or revision number, and the date it was prepared.

3.3.4.2 Table of contents. The table of contents should list the FSP elements, any appendices that are required to augment the FSP, tables, and figures.

3.3.4.3 Project background. This section of the FSP should be as specific as possible. Sufficient information should be included to permit a technical person unfamiliar with the project to evaluate the sampling and analytical approach presented. If items discussed here are in the project work plan, they do not need to be repeated, but they should be incorporated by reference. For those projects that do not require a work plan, refer to paragraph 1.7.1 for additional topics included within this section. This section of the FSP should include a description of the location, size, and important physical features of the site, such as ponds, lagoons, streams, and roads (a map showing the site location and layout would be helpful). A chronological site history including descriptions of the use of the site, complaints by neighbors, permitting, and use of chemicals should be provided. The historical data from previous sampling efforts at the site should be identified and summarized. An assessment of the quality of the historical data should be included as well as a discussion of problems previously encountered. The

effects of this information on the current project should also be discussed. This section should also describe the site problem to be resolved and the project approaches planned to work toward this resolution.

3.3.4.4 Project organization and responsibilities. This element of the FSP identifies key field personnel or organizations that are necessary for each field activity during the project. For remedial action and/or construction projects, this element will be expanded to include key personnel for all activities including project planning, since no overall project work plan is required. A table or chart showing the organization and lines of authority should be included. When specific personnel cannot be identified, the organization with the responsibility should be listed. The organization chart should also include all subcontractors and their key points of contact (POC). Separate organization charts for subcontractors may be referenced as needed. The organization chart should identify QC managers, including POCs of subcontractors, and should illustrate their relationship to other project personnel. The QC managers should be organizationally independent of the project management so that the risk of conflict of interest is minimized. This section of the FSP should also describe the responsibilities of all project field personnel, including sampling personnel, liaison between field and laboratory, and QC managers. It should designate responsibility for planning, coordination, sample collection, disposal of investigation-derived waste, and sample custody. This section should also identify any special training requirements and/or personnel certifications necessary to perform the project work.

3.3.4.5 Project scope and objectives. This section should identify the project activities planned, incorporating QA elements to be implemented to support those activities, any relevant regulatory standards, and the project schedule. EM 200-1-6 provides information on the QA elements that may be used for project chemical data quality assessment. Project QA procedures include laboratory validation, field and laboratory audits, PE samples, data validation procedures, etc. The project assessment activities employed should be discussed as they pertain to the general QA objectives identified for the project. Specific objectives of a sampling effort that describe the intended use of data should be clearly and succinctly stated. These objectives should satisfy not only the intended uses of the data, but also applicable regulatory standards or client preferences. This section of the FSP should also discuss special situations where site access may need to be obtained from private property owners, if applicable. In addition, an outline should be included of the project schedule based on client and regulatory requirements. Items listed on the project schedule should include project plan review periods, easement/permit periods, fieldwork, sample analysis, data management and validation, and project report writing.

3.3.4.6 Nonmeasurement data acquisition. This section of the FSP should describe those data needed from nonmeasurement sources. This may include information obtained from databases, literature, handbooks, local planning authorities, and other specific organizations. Information of this type may be needed to support risk assessment (local relevant or significant habitats, endangered species, future land uses, and well surveys); geological data (site bedrock formations, soil series); hydrogeological data (local or regional aquifers); meteorological data; data supporting modeling activities, etc.

3.3.4.7 Field activities by area of concern (AOC). This section of the FSP discusses the field activities planned for the work effort. For ease of review and referencing, it is recommended recommend that the SAP field activities be organized by area of concern (AOC) initially, with applicable matrices for that area managed as subsections.

3.3.4.7.1 Rationale/design. This section of the FSP discusses the rationale for each of the field activities. Each subsection that describes the matrices to be sampled within the AOC should include the

rationale behind the required number of field samples; the strategy (statistical, judgmental, and/or random basis) for selecting the particular sampling location (as discussed in Instruction C-1, Appendix C); a summary of the estimated or required number of field, background/upgradient, and field control samples; whether temporal changes can affect the representativeness of the sample; and the type of samples (composite or discrete). If a grid pattern is to be used to locate sample points, a discussion of the grid dimensions and layout should be included. Procedures should be defined for how sample locations are to be marked for future reference. As an alternative to the prescriptive approach of defining sample numbers and locations, is developing a decision logic that is used in conjunction with field analytical results to support onsite decision-making on the progress and direction of subsequent site sampling activities. Maps should be included to present site/AOC boundaries, any significant features, onsite buildings, ground water flow, existing monitoring wells, proposed sampling points, and any known contamination or plumes.

3.3.4.7.2 This FSP section should also discuss the analytical protocol and the rationale on how that relates to the site history and objectives of the work effort. Note any relevant action or decision levels. Also note when analytical protocols will change or differ within the same matrix, and the rationale for this approach. For instance, when based upon preliminary results, subsequent sample analytical protocols are abbreviated, or target compound lists are short-listed. The use of tables is suggested to present the information on sample analytical protocols, including appropriate analytical method numbers. Reference the QAPP portion of the SAP for details on method target analyte lists, sensitivity requirements (detection and quantitation limits), holding times, etc. Refer to Appendix I for details on analytical chemistry requirements. This FSP section should also list all field measurements that will be made during the project, noting the field analytical technologies, the protocols used, and the onsite decisions that the field data will support. Attach all field analytical method SOPs to the SAP. Identify the site-specific indicator compounds to be monitored, which samples will undergo field analyses, and how the data will be used / applied to a decision logic, or the effect on the sampling scheme this data will have. For instance, field screening data may direct samples for offsite definitive analysis, may define an appropriate low-level or high-level analytical method, or may be used to supplement a larger data set from which to support project decisions. Depending on the intended use of the field data, comparability to a definitive analysis may be an important factor and should be discussed with data users. Recommend performing bench or pilot studies, field trials, or establishing interim milestones early in the work effort to compare the field analytical results to definitive results to assess the correlation between the techniques and the viability of the field analytical technique. Further recommend that a percentage of field analyzed samples undergo redundant definitive analysis throughout the project to update the correlation, accuracy, and precision goals of the field analytical technique based on data's use. Verification should encompass samples that cover the entire range of contamination found onsite. Refer to Appendix H for details on field analysis technologies implementation. The discussion of field control samples should include the objective and rationale for these samples (how the data will be used), as well as the location and frequency for collecting/submitting these samples. This may include QA and QC duplicates, matrix spike/matrix spike duplicates, double-blind PE samples, equipment blanks, and/or trip blanks. This frequency may be expressed as a definitive number or percentage of the total number of samples collected. The sample locations may be identified qualitatively (e.g., the samples anticipated or noted as most contaminated) or by a specific area or sample location. Refer to Instruction G-2, Appendix G, for information on field control samples that may be used to assess field activities. It is recommended that all field samples and field control samples planned be summarized in tabular form. This table should indicate by sample location or area of concern the total number of samples for each matrix and associated QA/QC samples. For projects involving a large number of samples or analyses, it may not be possible to include all matrices in a single table. For such cases, two or more tables may be necessary. Table 3-3 is an example of a subsurface soil sample summary table.

Table 3-3
Sample Summary Table for Subsurface Soils

Sample Location	Sample Depth[1]	Sample Number (Primary Lab)	QC Sample Number (Primary Lab)	Associated Trip Blank Number (Primary Lab)	Associated Rinsate Blank (Primary Lab)	Sample Number (QA Lab)	Associated Trip Blank Number (QA Lab)	Associated Rinsate Blank (QA Lab)	EPA 8260[2]	EPA 8270[2]	EPA 6010[2]	EPA 8081[2]
T-1	4-6 ft bgs	T-1	-	TB-1	RB-1	-	-	-	X	X	X	X
T-2	6-8 ft bgs	T-2	-	TB-1	RB-1	-	-	-	X	X	X	X
T-3	2-4 ft bgs	T-3/S-1	T-6/S-1	TB-1	RB-1	T-6/S-1/QA	TB-1/QA	RB-1/QA	X	X	X	X
	4-8 ft bgs	T-3/S-2	-	TB-1	RB-1	-	-	-	X	X	X	-
	8-12 ft bgs	T-3/S-3	-	TB-1	-	-	-	-	X	-	-	-
T-4	6-8 ft bgs	T-4	-	TB-2	RB-1	-	-	-	X	X	X	X
T-5	2-4 ft bgs	T-5/S-1	T-7/S-1	TB-2	RB-1	T-7/S-1/QA	TB-2/QA	RB-1/QA	X	X	X	X
	4-8 ft bgs	T-5/S-2	-	TB-2	RB-1	-	-	-	X	X	X	-
	8-12 ft bgs	T-5/S-3	-	TB-2	-	-	-	-	X	-	-	-
T-11	6-8 ft bgs	T-11	-	TB-2	RB-1	-	-	-	X	X	X	X
T-14	6-8 ft bgs	T-14	-	TB-2	RB-2	-	-	-	X	X	X	X
T-23	2-4 ft bgs	T-23	-	TB-2	RB-2	-	-	-	X	X	X	X

Notes: 1. The spatial relationship of sample and blank numbers in the table is important. This design of the sample numbers themselves is not required. However, duplicate samples must be blind to the primary laboratory.
2. USACE policy does not require trip or rinstate blanks for soil sampling activities, but allows for them if project-specific considerations warrant.
3. This table should be prepared prior to field activities and included in the SAP. The table prepared after field activities would document any deviation from the plan.

[1] bgs = below ground surface.
[2] The most recently promulgated versions of these methods taken from EPA/SW-846 must be used.

3.3.4.7.3 Field procedures. This section of the FSP addresses the field procedures for sampling each matrix within an AOC and should include a description of the sampling equipment (including material it is constructed of), any special conditions that are required for the preparation or installation of that sampling equipment, field measurements, sample collection, sample handling, sample packaging (including sample containers and preservatives), and equipment decontamination. Sample handling procedures may encompass techniques for compositing subsamples (define number of subsamples, grid patterns, etc.), homogenizing solid matrices, splitting of replicate samples, filtering aqueous matrices, or the special handling of solid samples for volatile organic compounds (VOC) analysis. Any special care in sample handling to avoid cross-contamination or unnecessary loss or degradation of contaminants should be defined. The use of maps, figures, charts, flow diagrams, or tables is recommended to clearly delineate a sampling program. Provide tables for identification of appropriate sample container numbers, types, and sizes by matrix/chemical parameter; preservatives; and analytical holding times. Also note when two or more sample analyses may be combined into one sample container. Relevant SOPs should be included within the SAP to identify appropriate step-by-step procedures for sample acquisition, handling, and packaging. Refer to the tables and instructions/SOPs included within Appendices B and E for additional guidance on these subjects. This section of the FSP should also include field analysis procedures for calibration and analysis, the acceptance criteria, and the required actions based on the field results. The specifications and requirements for field instrumentation, including the initial and continuing calibration, calibration verification, maintenance and inspection schedules, any factors that limit use of the equipment, as well as the requirements for field data evaluation and reporting must be outlined in the FSP. Table 3-4 is an example of a field instrument calibration table that may be used on projects. Finally, each section should discuss any time constraints or other difficulties with sending samples to the laboratory. Specify contingencies in the event of delays and/or slippage in the schedule. Additional information on field activity procedures is presented in the following subsections and in Appendices C, D, and E by individual matrices or sample handling technique.

Table 3-4
Field Instrument Calibration Table

Instrument Manufacturer	Analyzer Name	Detector	Chemical Parameter[1]	Calibration Requirement[2]	Performance Checks[3]
--	--	Photoionization	Total Ionizable Hydrocarbons	Daily	None
--	--	Flame ionization	VOC	Per measurement	Weekly
--	--	Ultraviolet	SO_2	Weekly	Zero, Span, Drift, etc.
--	--	Foil	Dissolved O_2	Daily	Calibration Check

[1] Compound, Analyte, or Detector Response.
[2] Calibrator Source, such as Hexane, Reference Material, etc.
[3] Frequency of Zero, Span, Response Time, Calibration Check, etc.

3.3.4.7.4 Geophysics. This section of the FSP should include a discussion of the objectives of geophysical analysis and the techniques proposed to meet these objectives. It should also address general topics discussed in Paragraphs 3.3.4.7.1 and 3.3.4.7.2, as appropriate. The discussion should include the rationale used to delineate the study area and determine the spacing for the geophysical measurements. The proposed equipment, equipment calibration, and quality control procedures; preliminary testing of the method employed and early termination procedures; reporting requirements; delineation of the area to be investigated; data processing; and interpretation of the data should also be included. Refer to EM 1110-1-1802, Geophysical Exploration for Engineering and Environmental Investigations, for the required information covering geophysical surveys.

3.3.4.7.5 Soil gas survey. This section of the FSP should define the type of soil gas survey to be performed (active or passive); provide a discussion of the objectives of the survey and the rationale for selecting the location and frequency of soil gas samples; define the equipment and methods used for drilling, the materials (casing, screen, etc.), and the installation methods, sampling methods, documentation requirements, and any of the general topics discussed in paragraphs 3.3.4.7.1 and 3.3.4.7.2 above. Refer to Instruction C-8, Appendix C, for further information on soil gas sampling.

3.3.4.7.6 Ground water. This section of the FSP should include a discussion of the objectives for ground water sampling, monitoring well location rationale (e.g., to determine ground water flows, identify upgradient contaminant sources) and the rationale for screen depth placement. Methods for monitoring well installation should be discussed and/or relevant SOPs attached to the SAP, including design construction details, procedures for well development and well purging, obtaining a water level measurement or determining if free product is present, aquifer testing and any field measurements, defining required pumps, filters, drilling and sampling equipment decontamination procedures, and well development and/or purge water disposal requirements. Also, refer to paragraphs 3.3.4.7.1 and 3.3.4.7.2 for additional topics to be addressed. It is suggested that this section of the FSP include a table to summarize the well depths, casing diameters, screen intervals, etc., for new and existing monitoring wells to be sampled. Additional language should be presented within the FSP to discuss whether free product is anticipated and the effect that may have on the sampling event. Refer to EM 1110-1-4000 for required information on installation of ground water monitoring wells at HTRW sites and Instruction C-2, Appendix C, for more information on ground water sampling.

3.3.4.7.7 Ground water filtration. There are diverse views on the filtration of ground water before sample preservation for metals analysis. Many regulatory programs no longer accept filtered samples as representative of "dissolved metal" concentrations. Studies also indicate that the results from unfiltered samples may be affected more by well installation and development procedures than ground water contamination present. Options include filtering; not filtering; or the use of alternative sampling techniques (i.e., low-flow sampling techniques). The option selected should be based on the objectives of the specific project including the acquisition of samples representative of aquifer geochemistry and contamination, the intended use of the analytical data, comparability with previous site results, and the position of the regulators. Since metals that are attached to soil particles present during digestion will also be detected, higher metal concentrations are typical for the unfiltered ground water sample than for the filtered sample. However, carbon dioxide and oxygen concentrations may change over time once a ground water sample has been taken. This may lead to changes in pH and Eh, which in turn may lead to the precipitation of metal species. Therefore, delayed filtration may result in the precipitation of metals, which are then filtered, resulting in even lower dissolved metals concentrations. For this reason, filtration of samples, if specified, should be performed shortly after collection within the field, preferably using an in-line system, in lieu of having the laboratory perform this procedure. Field filtration procedures must be conducted prior to sample preservation (acidification), and both should be performed prior to sample shipment. Instructions C-2 and E-1, found in Appendices C and E, respectively, contain additional information on ground water sampling and filtration techniques. Refer to EPA 540/4-89/001 and Heidlauf and Bartlett (1993) for additional information on this topic. An alternative procedure to filtering samples is the use of a low-flow (minimal drawdown) sampling technique for sample acquisition (see EPA 540/S-95/504). This technique is the preferred method to obtain representative ground water samples, especially for redox sensitive analytes such as chromium. In addition to obtaining a sample that has both the dissolved and natural colloidal fraction of the ground water, the amount of purging and investigation-derived waste can be minimized. This method requires the use of an appropriate pump and a flow-through cell with a multiparameter probe (dissolved oxygen, pH, oxidation-reduction potential, temperature, total dissolved solids, and turbidity). There is also less agitation of the sampled media with

this technique; therefore it is a preferred method for the collection of volatile samples. Refer to Instruction C-2, Appendix C, for further information on low-flow sampling techniques.

3.3.4.7.8 Subsurface soil. This section of the FSP should discuss the objectives of subsurface soil samples and the rationale for soil boring locations, note discrete and/or composite sampling, and discuss any field analytical parameters to be measured. When compositing of samples is done, define the size and shape of the grid pattern and number of subsamples to be combined. This section should also discuss drilling methods, sampling methods for physical and chemical analyses, decontamination procedures of the drilling equipment, documentation and logging procedures, and the general topics discussed in paragraphs 3.3.4.7.1 and 3.3.4.7.2. Documentation of soil boring information is required in order to log facts into the Geospatial Data System as outlined within Instruction F-1, Appendix F. Additional information on subsurface soil sampling procedures is presented in Instruction C-6, Appendix C, and in EM 1110-1-1906.

3.3.4.7.9 Surface soil and sediment. This section of the FSP should discuss objectives of surface soil and/or sediment samples, the rationale for location and frequency of discrete and/or composite samples, all procedures required for collecting samples, and any applicable topics from paragraphs 3.3.4.7.1 and 3.3.4.7.2. When compositing of samples is done, define the size and shape of the grid pattern and number of subsamples to be combined. Instructions C-5 and C-6 in Appendix C contain additional information on sampling of sediments and surface soils.

3.3.4.7.10 Surface water. This section of the FSP should discuss the objectives and types of surface water or surface water runoff to be sampled, the rationale for the location and frequency of these samples, all procedures required for collecting surface water samples, and any applicable topics identified in paragraphs 3.3.4.7.1 and 3.3.4.7.2. Instruction C-3, Appendix C, contains additional information on the sampling of surface water and/or surface water runoff.

3.3.4.7.11 Other matrices. This section of the FSP should discuss the rationale for the location and frequency of samples from other matrices and the general topics discussed in paragraphs 3.3.4.7.1 and 3.3.4.7.2. Examples of other matrices include perimeter or point-source air samples, various media (solid, liquid, or gas) from remedial process waste streams, potable water supplies, surficial samples, bulk materials, etc. Appendices C and D contain information on sampling other matrices.

3.3.4.8 Field operations documentation. This section of the FSP should identify the records used to document all field operations. The FSP should also identify the records and schedule for those which require periodic submittal to a USACE representative. Project records may include the daily contractor QC reports, field logbook, field sheets, or other logs (i.e., boring logs, well installation diagrams, well development forms), any photographic records, and any field analytical records. This section should also address the sample documentation records, such as the sample numbering system, sample labels, tags, or sample field sheets, chain-of-custody forms, custody seals, and lab notification sheets. Corrections to documentation entries must be defined according to procedures identified in paragraph F-1, Appendix F. Include all proposed forms and tables within this section of the project FSP. Custody requirements for the samples should be defined, as applicable, during sample collection, transfer, and within the laboratory. Custody requirements should also be established for the final evidence papers of the project. This includes all originals of field documentation and laboratory reports. Also define records management practices, such as review procedures and record retention requirements. Other records may also be included within this section, such as SOPs, corrective action reports, any manifesting, bills of lading, waste profile forms, test pit logs, drum log sheets, etc. Refer to Instruction F-1, Appendix F, for additional information on documentation.

3.3.4.9 Sample packaging and shipping requirements. This section of the FSP should include a discussion of sample packaging and shipping requirements in accordance with U.S. Department of Transportation regulations. Identify all appropriate laboratories, noting addresses and points of contact; a schedule for submitting samples; the mode of sample transportation (e.g., overnight courier); and any manifesting requirements for the shipment. A checklist is recommended to verify completeness of sample shipment preparations. An example facsimile of a checklist for shipping HTRW samples is presented in Figure 3-2. It is recommended that the receiving laboratories also document the condition of field samples upon receipt at the laboratory. This enables verification of correct sample volumes, sample preservation, cooler temperature, chain-of-custody completeness and accuracy, and overall packaging techniques. An example facsimile of a cooler receipt documentation is presented in Figure 3-3. Another document beneficial to the laboratory by providing information on sample custody, preparatory/analysis methods, and data reporting requirements is the Laboratory Notification Checklist (LNC). It is recommended that the LNC be prepared and forwarded to the lab with the approved SAP, and a copy accompany the first shipment of incoming samples to the primary and referee (QA) laboratories. A facsimile of the LNC is presented in Figure 3-4. Instruction F-2, Appendix F, gives more information on sample packaging and shipping procedures; and sample receipt/log-in procedures are noted in paragraph I.5.1, Appendix I.

3.3.4.10 Investigation-derived wastes (IDW). This section of the FSP should discuss the procedures for collecting, labeling, storing, and disposing of the IDW. The procedures for assessing corresponding sample results or sampling the IDW to determine whether it is hazardous should be explained. Finally, the discussion should address how the sample results will be evaluated to determine disposal options for the IDW. It is important to note that disposal actions must be conducted with the concurrence of appropriate USACE technical personnel and that the final disposal decision must be agreed to by all parties.

3.3.4.11 Field assessment/three-phase inspection procedures. The contractor is required to ensure that quality is maintained throughout all field work by means of a three-phase control process (Engineer Regulation (ER) 1180-1-6 and Corps of Engineers Guide Specifications (CEGS) 01450 and 01451). Contractor quality control (CQC) phases (preparatory, initial, and follow-up) are performed onsite by a contractor-assigned QC officer whether or not a Government representative is present. The Contractor will summarize the activities of each CQC phase in the daily QC report. The CQC phases are performed for each definable feature of work. A definable feature is a task that is separate and distinct from other tasks and has separate control requirements. For example, the definable features of the sample collection task include, at a minimum, each matrix (air, water, soil, containerized waste, etc.). This section of the FSP should contain the contractor's detailed plans for implementing the CQC process, including identification of the CQC representative; listing of field equipment; description of activities during the phases; identification of the definable features of work; and generation of a sample table that will be used to match up primary and QA samples, as well as other field control samples required for the project. Paragraph G-1 in Appendix G contains additional information and checklists to help plan the preparatory and initial phases, and Table 3-3 is an example of a sample summary table. The equipment list noted may also be used by USACE QA personnel to support their preparatory phase inspection procedures of CQC. For USACE in-house projects, this section will include a list of the field equipment that will be required to perform the field activities and a sample table that correlates field samples to the appropriate field control (QA/QC) samples.

3.3.4.12 Nonconformance/corrective actions. The FSP should include corrective action procedures to be taken in the event a discrepancy is discovered by field personnel or during a desk or field audit, or the laboratory discovers discrepancies or problems. Typical discrepancies or problems include but are not limited to improper sampling procedures, improper instrument calibration procedures, incomplete or improper sample preservation, and problems with samples upon receipt at the laboratory.

SHIPPING CONTAINER CHECKLIST SUMMARY

ATTN.: Corps of Engineers Contractors

Failure to properly handle or document the Project samples could jeopardize the useability of the sample results and ultimately the project. Prior to sending this cooler to the Analytical Laboratory at the address shown below, please check the following items:

- Is the project clearly identified on the Chain-of-Custody (official project name, project location, project phase)? Is the United States Army Corps of Engineers project number from the Sampling and Analysis Plan clearly indicated on the Chain-of-Custody?
- Are all enclosed sample containers clearly labeled with waterproof (permanent) ink and enclosed in a plastic bag?
- Are the desired analyses indicated on the bottle labels and chain-of-custody? Are the metals defined on the Chain-of-Custody (e.g., metals = lead, cadmium, etc.)?
- Are the sample labels complete, including the identification of appropriate method numbers for both the preparatory and analysis procedures?
- Does the information on the Chain-of-Custody match the information on the sample container labels?
- Have you placed the Chain-of-Custody in a plastic bag and attached it to the inside of the cooler lid?
- Have the samples been properly preserved (acid or base and cooling to 4°C ± 2 °C)?
- Is there a Contractor point of contact including name and phone number clearly shown on the Chain-of-Custody?
- Is there sufficient ice (double bagged in zip-locks) or "blue ice" in the cooler? It is recommended that the samples be placed on ice as soon as possible after sampling and repacked on new ice in shipping cooler.

This is a partial list of the requirements for proper documentation and shipping of the environmental samples. Please refer to the Sampling and Analysis Plan for further details.

Figure 3-2. Shipping container checklist

COOLER RECEIPT FORM Contractor Cooler_____

LIMS#_____ QA Lab Cooler #_____

Number of Coolers_____

PROJECT:_____ Date received:_____

USE BOTTOM OF PAGE 2 OF THIS FORM TO NOTE DETAILS CONCERNING CHECK-IN PROBLEMS.

A. PRELIMINARY EXAMINATION PHASE: Date cooler was opened: _____
by (print)_____ (sign)_____

1. Did cooler come with a shipping slip (air bill, etc.)? ..... YES NO
   If YES, enter carrier name & air bill number here: _____
2. Were custody seals on outside of cooler? ..... YES NO
   How many & where _____, seal date: _____ seal name:_____
3. Were custody seals unbroken and intact at the date and time of arrival? ..... YES NO
4. Did you screen samples for radioactivity using the Geiger counter? ..... YES NO
5. Were custody papers in a plastic bag & taped inside to the lid? ..... YES NO
6. Were custody papers filled out properly (ink, signed, etc.)? ..... YES NO
7. Did you sign custody papers in the appropriate place? ..... YES NO
8. Was the project identifiable from custody papers? If YES, enter project name at the top of this form ..... YES NO
9. Were temperature blanks used? ..... YES NO
   Cooler Temperature_____(°C) Thermometer ID No._____
10. Have designated person initial here to acknowledge receipt of cooler: _____ (date) _____

(Continued)

Figure 3-3. Cooler receipt checklist (Note: LIMS = Laboratory Information Management System) (Continued)

B. LOG-IN PHASE: Date samples were logged in: _____
by (print) _____ (sign) _____

11. Describe type of packing in cooler: _____

12. Were all bottles sealed in separate plastic bags? ..... YES NO

13. Did all bottles arrive unbroken with labels in good condition? ..... YES NO

14. Were all bottle labels complete (ID, date, time, signature, preservative, etc.)? .... YES NO

15. Did all bottle labels agree with custody papers? ..... YES NO

16. Were correct containers used for the tests indicated? ..... YES NO

17. Were samples preserved to correct pH, if applicable? ..... YES NO

18. Was a sufficient amount of sample sent for tests indicated? ..... YES NO

19. Were bubbles absent in volatile organic analysis (VOA) samples? If NO, list VOA samples below ..... YES NO

20. Was the project manager called and status discussed? If YES, give details on the bottom of this form ..... YES NO

20. Who was called? _____ By whom? _____ (date)_____

Figure 3-3. (Concluded)

LABORATORY NOTIFICATION CHECKLIST

1. Project Name/Location: _____
2. Project Plans Title, Revision Number, and Date: _____
3. Contract Number: _____
4. Data Quality Objectives (DQOs) Summary (intended use of data): _____

_____

5. Lab Specific DQOs (Data quality indicators acceptance limits): _____

_____

6. Name of Person to be Contacted if there are Problems with the Sample Shipment:

_____

Phone Number: _____

FAX Number: _____

6. Name and Address of the Contract/QA Laboratories: _____
8. Project-Specific Requirements

Data Package Turn-Around Time: _____

Sample Retention Time Post-Analysis: _____

Sample Disposition Requirements: _____

MATRIX	SAMPLE NUMBERS	METHODS			REPORTING LIMITS (refer. SAP)
		PREP	CLEANUP	ANALYSIS	

9. Any Special Requirements (i.e., unusual target analytes, sample quick turnaround time (TAT)):

Figure 3-4. Laboratory notification checklist

3.3.5 Quality assurance project plan (QAPP). The following paragraph briefly describes the minimum requirements of a QAPP.

3.3.5.1 Title page. The title page should include the name of the document, (e.g., Phase I Remedial Investigation Quality Assurance Project Plan) and the date it was prepared. The contractor's laboratory QA manager or director should sign the title page if analytical services are provided. This will ensure that the laboratory is aware of the analytical methods employed and the measurement quality objectives for precision, bias, representativeness, completeness, comparability, and sensitivity. If the recommended signatures are difficult to acquire, suggest a statement be added that the approved SAP was provided to the appropriate parties and identify names of recipients and the date.

3.3.5.2 Table of contents. The table of contents should list the QAPP elements, any appendices that are required to augment the QAPP, and the titles of figures and tables. At the end of the table of contents the recipients of official copies of the QAPP should be listed.

3.3.5.3 Project laboratory organization and responsibilities. This element of the QAPP identifies key laboratory personnel or organizations that are necessary for each analytical activity during the study. All laboratories involved should be identified within this section, including the primary and referee (QA) laboratories. A table or chart showing the organization and lines of authority should be included. When specific personnel cannot be identified, the organization with the responsibility should be listed. The organization chart should also include all subcontractors and their key points of contact. Separate organization charts for subcontractors may also be needed. The organization chart should identify QA managers, including those of subcontractors, and should illustrate their relationship to other project personnel. The QA managers should be organizationally independent of project management so that the risk of conflict of interest is minimized. Requirements for the contractor's laboratory and laboratory personnel are located in Appendix I.4.3.

3.3.5.4 Data assessment organization and responsibilities. This element of the QAPP identifies personnel or organizations that will be performing data assessment activities. Applicable duties of personnel should be identified, and may include data review, verification, and/or validation procedures. A table or chart showing the organization and lines of authority and communication should be included. Refer to EM 200-1-6 for further information on data assessment procedures available.

3.3.5.5 Data quality objectives. Data quality objectives should be identified within this section to define the intended use of the data, and any specific conditions, criteria, or limits to be applied to the data based upon this use. Specific information to be included is outlined in the following sections.

3.3.5.5.1 Data use background. This section should highlight project-specific data needs that have been identified for the project, short-term decisions that will be made during the project planning phase, and long-term decisions that will be made prior to project closeout. A brief summary of the type of samples (media) and analyses (screening versus definitive, and applicable chemical parameters) that will be required to meet the data needs should also be included in this section. Refer to Sections I.3.1 and I.3.1.2 for a discussion of background information to be included in this section of the QAPP.

3.3.5.5.2 Measurement quality objectives for chemical data measurement. This section should describe the QA elements to be applied to the project to ensure proper CDQM. In addition, measurement quality objectives (MQOs) are established for key data quality indicator terms (precision, accuracy, sensitivity, etc.) to evaluate these QA elements for each matrix and analytical method. Refer to EM 200-1-6 for information on QA elements; and Appendix I for discussions on data quality indicators, MQOs,

and baseline method performance objectives. A cooperative effort should be undertaken by USACE, the lead agency, the contractor, and the laboratory staff when defining the project-specific MQOs required for the data. They should be based on a common understanding of the intended use of the data, available laboratory procedures, and available resources. However, these objectives should be defined in terms of project requirements, not in terms of the capabilities of the test methods. In addition, sensitivity (detection limits) expressed within a method are often based on a reagent water matrix and may not be achievable within an environmental matrix. However, when the requirements for the project are defined, the quality requirements established by the data user, the complexity of the media to be analyzed, the potential for interferences, etc., must be considered. This section should also address whether precision is being applied to field activities, to assess the laboratory performance, or both. Separate precision goals should be established for grab samples of a heterogeneous media (i.e., soil VOC samples). However, when sample handling techniques used are the same (i.e., field and laboratory duplicates of aqueous or homogenized soils), a separate criterion is not necessary. Because precision and bias can be measured in various ways, the calculation to be used should be identified. Completeness goals must be defined quantitatively, and whether it is being applied to individual samples, or for the work effort as a whole. Refer to Appendix I for further information on the remaining parameters of representativeness, comparability, and sensitivity. The use of tables to define the appropriate MQOs for each analytical method is recommended. Appendix I includes summary tables with default values that may be used verbatim, or changed to reflect project-specific DQOs. Due to the limited applications where the implementation of an EPA standard method is mandatory, these MQOs must be specified directly within the QAPP verbatim or by reference to ensure application to the project sample analyses. If the project MQOs are unattainable by available methods, either the methods or test plan must be compensated for these deficiencies. The following statements are examples of descriptions for precision and bias:

- Field precision objectives for the soil VOC samples are presented as relative percent difference of field duplicates and are presented within the attached tables. Any exceedance of the MQO values will trigger corrective action requirements as noted in Appendix I.

- Bias objectives for organic compounds are given as a percent recovery range of all target analytes within the laboratory control sample and matrix spikes, and as surrogate compounds in all field and QC samples. Bias goals are established by calculation of percent recovery, and are presented within the attached tables. Any exceedance of the MQO values will trigger corrective action requirements as noted within Appendix I.

3.3.5.6 Sample receipt, handling, custody, and holding time requirements. This section should identify the requirements for sample receipt condition verification, sample storage and/or handling requirements, any intralaboratory custody requirements, and analytical parameter holding times. In addition, all notifications, customer correspondence, and corrective actions for incoming samples must be thoroughly documented and available for review.

3.3.5.7 Analytical procedures. This section of the QAPP identifies the appropriate analytical test methods to be used for each environmental sample. The applicability of an individual method will be dependent upon the regulatory authority and the level of data quality required to support the data needs and decisions of the project. Applicable regulations that mandate the use of certain methods for any of the sample matrices and parameters listed in the project description should be specified. The use of tables is recommended to present this information clearly. It is suggested that the table be divided into each AOC, with proposed chemical parameters (including preparatory and analytical method numbers) defined for each matrix to be sampled within that area. Analytical chemistry requirements are presented in Appendix I.

3.3.5.7.1 Preventive maintenance. This section of the QAPP should discuss the laboratory's preventive maintenance plan that will be implemented to minimize downtime of laboratory instruments. If appropriate for the project, include a reference to Appendix I in order to apply the requirements specified therein.

3.3.5.7.2 Calibration procedures and frequencies. This section of the QAPP discusses the calibration procedures that are to be used by the contractor's laboratory. Issues that should be addressed in this section include defining the number and concentration of calibration standards to be used, the calibration range, and the procedures used to establish and verify the calibration of the laboratory's instruments. If appropriate for the project, include a reference to Appendix I in order to apply the requirements specified therein.

3.3.5.7.3 Laboratory QC procedures. This section of the QAPP identifies the specific internal QC measures to be used by the laboratory when performing the analytical tests. Type and frequencies of specific QC samples performed by the laboratory are dependent upon the specified analytical method. Internal QC methods require performance on a sample batch basis and include analyses of method blanks, laboratory control samples, and actual environmental samples as duplicates, matrix spikes, and matrix spike duplicates. Additional QC is incorporated into the analytical sequence. A more detailed discussion of internal QC procedures is presented in Appendix I. If appropriate for the project, include a reference to Appendix I in order to apply the requirements specified therein.

3.3.5.7.4 Performance/system audits. This section of the QAPP describes the performance and system audits that will be performed onsite and at the contractor's laboratory. The laboratory audits are typically conducted internally by the laboratory QA staff, as well as by external agencies. USACE performs laboratory audits in conjunction with the laboratory validation process. District personnel are encouraged to perform precontract or preaward system audits of the laboratory to ensure that proper communication and awareness of project DQOs are in place.

3.3.5.7.5 Nonconformance/corrective actions. This section of the QAPP addresses corrective actions that must be implemented if MQOs are not met. The QAPP should discuss corrective action procedures that will be implemented if problems are observed with incoming samples, sample holding times, instrument calibration procedures, specified detection and quantitation limits, or internal QC samples. Corrective actions may include resampling, reanalyzing samples, or auditing laboratory procedures. The QAPP should identify persons responsible for initiating these actions. It should also contain procedures for identifying and documenting corrective actions and procedures for reporting and follow-up of corrective actions. A more detailed discussion of corrective action procedures is contained in Appendix I. If appropriate for the project, include a reference to Appendix I in order to apply the requirements specified therein.

3.3.5.8 Data reduction/calculation of data quality indicators. This section of the QAPP should discuss how data are reduced by the laboratory and define how the precision, bias, sensitivity parameters (detection, quantitation, and reporting limits), and completeness goals are to be calculated from the project data. Data reduction procedures must be summarized and the persons responsible for data reduction must be identified. A more detailed discussion of the methods used to calculate these data quality indicators is presented in Appendix I.

3.3.5.9 Laboratory operations documentation. This section of the QAPP discusses the data reporting procedures for the project. The reporting package format, contents, reporting schedule, data archival, and records retention requirements should be identified. Any electronic data deliverables

format and technical content must be identified here also. Refer to Appendix I for information on the potential content requirements for screening, definitive, performance-based, and comprehensive data packages. For projects that involve a substantial number of samples, or projects that require continued monitoring, the use of interim data deliverables for reporting is recommended. These deliverables should be submitted after a proposed milestone instead of at the project completion.

3.3.5.10 Data assessment procedures. This section of the QAPP discusses the data review, verification, or validation process that is required to assure the validity of the data. It also discusses the DQO reconciliation process and the effect that it has on the final assessment of project completeness. Refer to EM 200-1-6 for further information on data assessment procedures.

3.3.5.10.1 Data QC review. The data review process should be discussed. Personnel who will have the various roles and responsibilities must be addressed in detail. The use of diagrams is suggested to outline the flow of the data as the project progresses.

3.3.5.10.2 Data verification/validation. The project requirements for data verification or validation must be defined. Terms used (i.e., validation) and procedures for implementation must be clearly defined in order to avoid any confusion on the anticipated level of effort for this task. A discussion of these terms, i.e., data review, validation, etc., and their requirements are presented in EM 200-1-6. Based on the project data use, implementation of an independent validation of the data may be necessary. Provide details on the percentage of data undergoing the validation process, and the procedures to follow. Refer to other USACE guidance for procedures for performance-based review, and validation procedures to encompass a manual full data validation for common EPA SW-846 methods.

3.3.5.10.3 DQO reconciliation. DQO reconciliation procedures should be identified. Details must be included that identify the personnel responsible for these tasks (contractor and/or USACE), the procedures to follow, any statistical tests to be employed, etc. DQO reconciliation is generally discussed in EM 200-1-2 and EM 200-1-6. Further guidance on this subject may be obtained from EPA QA/G-9.

3.3.5.10.4 Project completeness assessment. Completeness assessment for the project is also discussed in EM 200-1-6. Procedures for the contractor as well as for USACE anticipated for the project should be defined within this section of the QAPP.

3.3.6 Appendices. References, standard forms, and a list of abbreviations and acronyms, for example, should be included in appendices.

Chapter 4
Sampling and Analysis Protocols

4.1 General

This chapter provides guidance to USACE personnel and USACE contractors for using the sampling and analytical instructions in the appendices to this manual and for developing project-specific instructions if project-specific characteristics make it impractical to use the sampling and analytical instructions found in these appendices. Issues other than those identified in the general SAP format requirements found in Chapter 3 may have to be included in the SAP to meet project-specific regulatory requirements. To meet project-specific protocols and satisfy any additional requirements, additional field and analytical SOPs and references have been included in Appendix A. General guidance for developing additional site-specific instructions has been included in this chapter. With respect to sampling and analytical protocols, the information provided herein may be used to prepare the SOW for the project or to prepare the SAP. In some instances, data collection activities will occur that are not covered by this manual. The references in Appendix A and the discussion in paragraph 4.4 may be useful under these circumstances.

4.2 Selecting Sampling and Analytical Instructions

As discussed in paragraph 2.3, selection of sampling and analytical protocols for a specific site is dependent upon the site constraints, data needs and data quality objectives, and sampling strategies for the various media. After an analysis of these factors has been completed, sufficient information should exist to select appropriate sampling and analytical instructions from the appendices in this manual. As discussed in EM 200-1-2, sampling and analytical options and appropriate SOPs or instructions should be selected after consideration of the following criteria: schedule, regulatory, technical (effectiveness and implementability), and budget. Guidance to follow during the selection process is provided in the following subsections.

4.2.1 Sampling instructions. Information gathered from Steps 1 and 2 discussed in paragraph 2.3 should be used to identify applicable sampling protocols from the instructions in the appendices. An analysis of the constraints at the site will provide information needed to propose sampling locations and sampling procedures. This analysis should consider the media to be sampled, the types of contaminants, and the physical characteristics of the site. Project resource constraints will also be a factor. An analysis of data needs and DQOs will identify applicability of filtration, compositing, and homogenization procedures and field QC requirements. Sampling strategies should also be reviewed to determine the location and frequency of samples. After this information has been reviewed, appropriate sampling method options may be developed from the instructions in the appendices. If the instructions in the appendices do not contain an appropriate sampling method, alternative methods may be developed using the relevant references in Appendix A and the procedures described in paragraph 4.4.

4.2.2 Analytical instructions. The information needed to properly select an analytical instruction can be obtained from following Steps 1 and 2 of paragraph 2.3. Analysis of the constraints at the site provides information about the sample matrix, measurement parameters, and regulatory and customer preferences in regard to the type of analytical method to be used. A review of the data needs may define additional sample handling procedures, including homogenization and subsampling requirements, detection and quantitation limit requirements, instrumentation requirements, and appropriate analytical methods. After this information has been reviewed, appropriate analytical options may be identified from the instructions in Appendix I. If the instructions identified in Appendix I do not contain an

appropriate analytical method, the relevant references in Appendix A and the procedures in paragraph 4.4 may be used to develop additional instructions.

4.3 Additional Standard Operating Procedures

If the appendices do not contain appropriate sampling and analytical protocols, it will be necessary to develop additional instructions. Paragraphs 2.3 and 4.2 of this manual should be consulted when deciding if other instructions need to be developed. Paragraph 4.4 discusses the methodology for developing instructions. The references in Appendix A contain information that may be used to develop additional instructions.

4.4 Development of Project-Specific Protocols

As previously discussed, it may be necessary to develop sampling and analytical protocols other than those identified in the appendices. Additional instructions may be required for a myriad of reasons: client preferences, regulators' preferences, unusual site conditions, budget considerations, etc. If it is determined that new sampling and analytical protocols need to be developed, or protocols other than those found in the appendices are preferred, then this section and the references in Appendix A can be used to develop the new protocols. However, it is important that the new instructions are able to satisfy data needs and DQOs as well as satisfying scheduling, regulatory, technical, and budget criteria. Guidance for developing new instructions follows. Additional information can be found in several of the references in Appendix A, especially EPA/600/2-80/018, EPA 600/4-79/020, EPA SW-846, and EPA/540/P-87/001.

4.4.1 Sampling instructions. The following list is a template that may be used as an outline to develop new sampling instructions. The references in Appendix A provide additional guidance.

(1) Scope and purpose.

(2) Definitions.

(3) Applicability.

(4) Sample locations.

(5) Applicable sampling strategies (discrete/composite: random, judgmental, stratified, etc.).

(6) When filtration is applicable (sampling for dissolved metals).

(7) When homogenization is applicable (sampling solid media).

(8) Method specified in entirety (step-by-step presentation).

(9) Field QC requirements (all field duplicates, all QA splits, trip blanks, background samples, highly contaminated media rinsates).

(10) Split-sample techniques/deviations from normal protocol.

(11) Preservation techniques (cool, acid preservation, base preservation, chlorine binding).

(12) Field measurements.

(13) Miscellaneous considerations.

4.4.2 Analytical instructions. The following list is a template that may be used as an outline to develop new analytical instructions. The references in Appendix A provide additional guidance.

(1) Title/Signature/Effective Date page

(2) Scope and application, including applicability to various matrices and discrete/composite procedures.

(3) Method summary

(4) Sample preservation

(5) Containers, handling, and storage

(6) Interferences and potential problems

(7) Equipment and apparatus

(8) Reagents and solutions

(9) Procedures

- Applicable sample preparation and cleanup procedures.
- Any applicable special sample handling requirements.
- Analytical method specified in entirety (step-by-step presentation).
- Instrumentation requirements.

(10) Calculations

(11) QC requirements for second-column confirmation, and/or the analysis of surrogates, matrix spikes, internal standards, blanks, laboratory control samples. All QC elements should define appropriate measurement quality objectives (MQOs) for appropriate data quality indicators (i.e., precision and bias).

(12) Corrective actions

(13) Data evaluation

(14) Method detection limit studies/sensitivity assessment

(15) Analyst experience requirements

(16) Health and safety

(17) Sample disposal

(18) References

(19) Definitions

(20) Example forms

Appendix A
References

A-1. Required Publications

40 CFR 50
National Primary and Secondary Ambient Air Quality Standards, Measurement Methods, 40 CFR Part 50, Appendixes A through J.

40 CFR 51
Recommended Test Methods for State Implementation Plans (SIP), 40 CFR Part 51, Appendix M

40 CFR 60
New Source Performance Standards (NSPS) Methods, 40 CFR Part 60, Appendix A
Continuous Emission Monitors (CEM) Performance Specifications, 40 CFR Part 60, Appendix B

40 CFR 61
National Emission Standards (NES) for Hazardous Air Pollutants, 40 CFR Part 61, Appendix B

40 CFR 261
Toxicity Characteristic Leaching Procedure, 40 CFR Part 261, Appendix U

40 CFR 266
Methods Manual for Compliance With the BIF Regulations, 40 CFR Part 266, Appendix IX

40 CFR 761
Polychlorinated Biphenyls (PCBs) Manufacturing, Processing, Distribution in Commerce, and Use Prohibitions

49 CFR 171
General Information, Regulations, and Definitions

49 CFR 172
Hazardous Materials Table, Special Provisions, Hazard Materials Communications, Emergency Response Information and Training Requirements

49 CFR 173
Shippers - General Requirements for Shipment and Packaging

49 CFR 174
Carriage by Rail

49 CFR 175
Carriage by Aircraft

49 CFR 176
Carriage by Vessel

49 CFR 177
Carriage by Public Highway

49 CFR 178
Specifications for Packaging

ER 1110-1-263
Chemical Data Quality Management for Hazardous Waste Remedial Activities

ER 1110-1-8156
Policies, Guidance, and Requirements for Geospatial Data and Systems

EP 200-1-2
Process and Procedures for RCRA Manifesting

EP 715-1-2
A Guide to Effective Contractor Quality Control (CQC)

EP 1110-1-21
Air Pathway Analysis for the Design of Hazardous, Toxic, and Radioactive Waste (HTRW) Remedial Action Projects

ETL 1110-1-175
Practical Aspects of Geostatistics in Hazardous, Toxic, and Radioactive Waste Site

EM 200-1-1
Validation of Analytical Chemistry Laboratories

EM 200-1-2
Technical Project Planning (TPP) Process

EM 200-1-6
Chemical Quality Assurance for HTRW Projects

EM 1110-1-2909
Geospatial Data and Systems

EM 1110-1-4000
Monitoring Well Design, Installation, and Documentation at Hazardous, Toxic, and Radioactive Waste Sites

American National Standards Institute/American Society for Quality Control 1994
American National Standards Institute/American Society for Quality Control. 1994. "American National Standard Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs," ANSI/ASQC E-4.

American Public Health Association 1995
American Public Health Association. 1995. *Standard Methods for the Examination of Water and Wastewater,* American Public Health Association (APHA), American Water Works Association (AWWA), and the Water Pollution Control Federation (WPCF), 19th Edition, Washington, DC.

American Society for Testing and Materials
American Society for Testing and Materials. "Annual Book of ASTM Standards," West Conshohocken, PA, to include the following standards.

D 1452 Standard Practice for Soil Investigation and Sampling by Auger Borings

D 1586 Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils

D 1587 Standard Practice for Thin-Walled Tube Sampling of Soils

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Appendix B
Preservatives, Holding Times, and Sample Containers

Table B-1
Preservatives, Holding Times, and Sample Containers for SW-846 Methods[1]

	Preservatives[3]		Holding Time		Containers[4]	
Parameter[2]	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Volatile Organics[5] (VOCs) Including: 8260 - VOCs 8021 - Aromatic VOCs 8021 - Halogenated VOCs 8015 - Nonhalogenated VOCs 8032 - Acrylamide	No Residual Chlorine present Adjust pH to < 2 with H_2SO_4, HCl, or solid sodium bisulfate ($NaHSO_4$). Cool to 4 °C.	Low-level soil [6] Add ~5 g soil to 40-mL VOA vial pre-preserved with 1 g of $NaHSO_4$/5 mL water.	14 days	Low/High Level 14 days	3 × 40 mL glass VOA vial, PTFE septa cap	3 × 40 (or 60) mL glass VOA vial (with stir bar for low-level soil), PTFE septa cap[7]

N/A - Not Applicable.
[1] Additional standard method manuals may be applicable. Refer to individual methods employed for details.
[2] Major chemical parameter headings (i.e., Volatile Organics, Extractable Organics, Metals, etc.) are given in boldface type. When applicable, subset chemical parameters and method numbers have been added to clarify that the same preservative, container, and holding time requirements apply. When multiple chemical parameters or chemical parameter subsets are needed, the specified number of sample containers must be submitted to the laboratory for each chemical analysis requested.
[3] Reagents should be of appropriate grade or quality as identified within the analytical method.
[4] All containers must have PTFE (Teflon)-lined seals (Teflon-lined septa for volatile organic analyte (VOA) vials).
[5] VOC section is segregated based on preparatory procedures employed. Refer to SW-846 Method 5000 for details on determinative methods application to appropriate preparatory procedures.
[6] Refer to SW-846 Method 5035 for additional preservation options.
[7] Actual number of sample containers necessary for each location is based on project information. Refer to Instruction E-5 in Appendix E for additional details.

Table B-1 (Continued)

Parameter[2]	Preservatives[3] Aqueous Liquid	Preservatives[3] Solid	Holding Time Aqueous Liquid	Holding Time Solid	Containers[4] Aqueous Liquid	Containers[4] Solid
VOCs (Cont.) 8033 - Acetonitrile 8315 - Carbonyl Compounds[8] Prepared by: 5030 - Purge and trap (aqueous) 5035 - Closed system purge and trap (solid)	Residual Chlorine present Collect sample in a 125-mL container, prepreserved with 4 drops of 10% sodium thiosulfate ($Na_2S_2O_3$) solution. Gently swirl to mix, and transfer to 40-mL VOA vials. Adjust pH to < 2 with H_2SO_4, HCl, or solid $NaHSO_4$. Cool to 4 °C, no headspace.	High-level soil Add ~5 g soil to 40- (or 60-) mL VOA vial prepreserved with 10 mL methanol. Concentrated waste[9] Collect sample in a 40- (or 60-) mL VOA vial with minimal headspace. Cool to 4 °C. EnCore™ Sampler (or similar) Collect 5-g samples according to manufacturer's instructions.		EnCore™ Sampler 48 hours		3 - EnCore™ Sampler (or similar)[7]
Prepared by: 5021 - Automated Headspace	N/A	Soil only Add ~2 g soil to 22-mL soil vial. Cool to 4 °C. Soil/Matrix Modifier[10] Add ~2 g soil to 22-mL soil vial prepreserved with 10 mL matrix modifier. Cool to 4 °C. Soil/Water Add ~2 g soil to 22-mL soil vial prepreserved with 10 mL water. Cool to 4 °C.	N/A	14 days	N/A	2 × 22 mL glass soil headspace vial, PTFE-lined septa with crimp or screw-top cap[7]
Prepared by: 5032 - Vacuum Distillation	Same as VOC - purge and trap.	Cool to 4 °C. No headspace.	14 days	14 days	2 × 40 mL glass vial, PTFE septa cap	2 × 125 - mL CWM[11]

[8] Samples must be derivatized and extracted within 3 days of collection and analyzed within 3 days of extraction.
[9] Refer to SW-846 Method 5035 for additional preservation options for oily waste.
[10] Refer to SW-846 Method 5021 for details on the matrix modifying solution needed.
[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).

Table B-1 (Continued)

Parameter[2]	Preservatives[3] Aqueous Liquid	Preservatives[3] Solid	Holding Time Aqueous Liquid	Holding Time Solid	Containers[4] Aqueous Liquid	Containers[4] Solid
Nonpurgeable Water-Soluble VOCs Prepared by: 5031 - Azeotropic Distillation	Same as VOC - purge and trap.	Cool to 4 °C, No headspace.	14 days	14 days	2 × 40 mL glass vial, PTFE septa cap	2 × 125 - mL CWM[11]
VOCs Prepared by: 3585 - Solvent Dilution	N/A	Oily waste Cool to 4 °C.	N/A	14 days	N/A	125-mL CWM[11]
VOCs Including: 8031 - Acrylonitrile 8316 - Acrolein, Acrylamide, Acrylonitrile	Adjust pH to 4-5 with H_2SO_4, HCl, or solid $NaHSO_4$. (Cool to 4 °C.	N/A	14 days	N/A	2 × 40 mL glass vial, PTFE septa cap	N/A
Extractable Organics Including: 8270 - Semivolatile Organics 8041 - Phenols[13] 8061 - Phthalate Esters 8070 - Nitrosamines[14] 8081 - Organochlorine Pesticides[14] 8082 - Polychlorinated Biphenyls[14] 8091 - Nitroaromatics / Cyclic Ketones[14] 8100 - PAHs[14] 8111 - Haloethers[14] 8121 - Chlorinated Hydrocarbons 8151 - Chlorinated Herbicides 8310 - PAHs 8321 - Nonvolatile Organics 8325 - Nonvolatile Organics[12] 8330 - Nitroaromatics and Nitramines (explosives)[14] 8331 - Tetrazene [14] 8332 - Nitroglycerine	No Residual Chlorine present Cool to 4 °C. Residual Chlorine present Add 1 mL 10% sodium thiosulfate ($Na_2S_2O_3$) solution per liter water. Cool to 4 °C.	Cool to 4 °C.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17] per chemical parameter	250 mL CWM[11,18,19]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[12] Extracts must be stored at -10°C and in the dark until analysis.
[13] Extracts should be methylated within 48 hours of extraction, and analyzed immediately thereafter.
[14] Extracts must be stored at 4°C and in the dark until analysis.
[15] 7 days until extraction/analyzed within 40 days after extraction.
[16] 14 days until extraction/analyzed within 40 days after extraction.
[17] AG - Amber Glass bottle(s) with Teflon-lined cap(s).
[18] The subset chemical parameters under Extractable Organics may be collected in the same container for soils.
[19] High-concentration waste samples do not need cooling.

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Extractable Organics 8141 - Organophosphate Pesticides	Adjust to 5 < pH < 9 with H_2SO_4 or NaOH. Cool to 4 °C.	Cool to 4 °C.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17]	250 mL CWM[11]
Extractable Organics 8318 - N-Methylcarbamates	Adjust pH to 4-5 with 0.1 N chloroacetic acid. Cool to 4 °C. Store in dark.	Cool to 4 °C. Store in dark.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17]	250 mL CWM[11]
Extractable Organics 8280 - Dioxins / Furans 8290 - Dioxins / Furans	No Residual Chlorine present If sample pH is greater than 9, adjust to pH 7-9 with sulfuric acid (H_2SO_4) Cool to 4 °C. Store in dark.	Cool to 4 °C. Store in dark.	30d/45d[20]	30d/45d[20]	2 × 1 L AG[17]	250 mL CWM[11]
	Residual Chlorine present Add 80 mg sodium thiosulfate ($Na_2S_2O_3$) per liter water. If sample pH is greater than 9, adjust to pH 7-9 with sulfuric acid (H_2SO_4). Cool to 4 °C. Store in dark.					
Extractable Organics 8131 - Analine / selected derivatives	No Residual Chlorine present Adjust pH to 6-8 with H_2SO_4 or NaOH. Cool to 4 °C.	Cool to 4 °C.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17]	250 mL CWM[11]
	Residual Chlorine present Add 35 mg sodium thiosulfate ($Na_2S_2O_3$) per ppm chlorine per liter water. Adjust pH to 6-8 with H_2SO_4 or NaOH. Cool to 4 °C.					

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[15] 7 days until extraction/analyzed within 40 days after extraction.
[16] 14 days until extraction/analyzed within 40 days after extraction.
[17] AG - Amber Glass bottle(s) with Teflon-lined cap(s).
[20] 30 days until extraction/analyzed within 45 days after extraction.

(Sheet 4 of 9)

Table B-1 (Continued)

	Preservatives[3]		Holding Time		Containers[4]	
Parameter[2]	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Metals (Except Chromium (VI) & Mercury)	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	1 L HDPE[21]	250 mL CWM[11,22]
Mercury	HNO_3 to pH < 2.	Cool to 4 °C.	28 days	28 days	250 mL HDPE[21] or glass	250 mL CWM[11]
Chromium (VI)	Cool to 4 °C.	Cool to 4 °C.	24 hours	24 hours	250 mL HDPE[21]	250 mL CWM[11]
Miscellaneous						
Acidity	Cool to 4 °C.	N/A	48 hours	N/A	250 mL HDPE[21]	N/A
Alkalinity	Cool to 4 °C.	N/A	48 hours	N/A	250 mL HDPE[21]	N/A
Ammonia	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	14 days	N/A	1 L HDPE[21]	N/A
Biochemical Oxygen Demand (BOD)	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	48 hours	N/A	2 L HDPE[21]	N/A
Biochemical Oxygen Demand (Carbonaceous)	Cool to 4 °C.	N/A	48 hours	N/A	2 L HDPE[21]	N/A
Bromide	Cool to 4 °C.	N/A	28 days	N/A	250 mL HDPE[21]	N/A
Chemical Oxygen Demand	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	28 days	N/A	125 mL HDPE[21]	N/A
Chloride	None Required.	None Required.	28 days	28 days	125 mL HDPE[21]	125 mL CWM[11]
Chloride (Total Residual)	None Required.	N/A	ASAP	N/A	500 mL HDPE[21]	N/A
Coliform, Fecal & Total	Cool to 4 °C (add 0.1 mL of 10% $Na_2S_2O_3$ if residual Cl present).	Cool to 4 °C.	6 hours	6 hours	120 mL HDPE[21]	125 mL CWM[11]
Color	Cool to 4 °C.	N/A	48 hours	N/A	125 mL HDPE[21]	N/A
Common Ions	Cool to 4 °C.	Cool to 4 °C.	28 days	28 days	1 L glass	250 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[21] HDPE - High-Density Polyethylene bottles.
[22] Metals and cyanide may be collected in the same container for soils.

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Miscellaneous (Cont.) Cyanide (Total & Amenable)	Cool to 4 °C and adjust pH > 12 with 50% NaOH If Oxidizing Agents are present: Cool to 4 °C. Add 5 mL 0.1 N $NaAsO_2$ per liter water or 0.06 g ascorbic acid per liter water. Adjust pH > 12 with 50% NaOH.	Cool to 4 °C.	14 days	14 days	1 L HDPE[21]	250 mL CWM[11, 22]
Dissolved Oxygen, Winkler Method	Fix on Site/Store in dark.	N/A	8 hours	N/A	300 mL BOD bottle.	N/A
Fecal Streptococci	Cool to 4 °C (0.008% $Na_2S_2O_3$ if residual Cl present).	N/A	6 hours	N/A	250 mL HDPE[21]	N/A
Fluoride	Cool to 4 °C.	N/A	28 days	N/A	500 mL HDPE[21]	N/A
Hardness	HNO_3 or H_2SO_4 to pH < 2.	N/A	6 months	N/A	250 mL HDPE[21]	N/A
Hydrogen Ion (pH)	None Required.	Cool to 4 °C.	24 hours	ASAP	60 mL HDPE[21]	125 mL CWM[11]
Kjeldahl and Organic Nitrogen	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	28 days	N/A	1 L HDPE[21]	N/A
Nitrate	Cool to 4 °C.	Cool to 4 °C.	48 hours	48 hours	250 mL HDPE[21]	250 mL CWM[11]
Nitrate-Nitrite	Cool to 4 °C, H_2SO_4 to pH < 2.	Cool to 4 °C.	28 days	28 days	250 mL HDPE[21]	250 mL CWM[11]
Nitrite	Cool to 4 °C.	N/A	48 hours	N/A	125 mL HDPE[21]	N/A
Oil & Grease	Cool to 4 °C and add 5 mL 1:1 (HCl).	Cool to 4 °C.	28 days	28 days	2 - 1 l glass	250 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[21] HDPE - High-Density Polyethylene bottles.
[22] Metals and cyanide may be collected in the same container for soils.

(Sheet 6 of 9)

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Miscellaneous (Cont.) Organic Carbon, Total (TOC)	Adjust pH to < 2 with H_2SO_4, HC1, or solid sodium bisulfate ($NaHSO_4$). Cool to 4 °C. Store in dark.	Cool to 4 °C.	28 days	28 days	125 mL HDPE[21]	125 mL CWM[11]
Orthophosphate	Filter immediately. Cool to 4 °C.	N/A	48 hours	N/A	125 mL HDPE[21]	N/A
Phenolics	Cool to 4 °C and adjust to pH < 4 with H_2SO_4.	Cool to 4 °C.	28 days	28 days	1 L BR[23]	250 mL CWM[11]
Phosphorus (Elemental)	Cool to 4 °C.	N/A	48 hours	N/A	1 L BR[23]	N/A
Phosphorus (Total)	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	28 days	N/A	125 mL HDPE[21]	N/A
Radiological Test, Gross Alpha	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	2 L HDPE[21]	250 mL HDPE[21]
Radiological Test, Gross Beta	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	2 L HDPE[21]	250 mL HDPE[21]
Radiological Radium (Total)	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	2 L HDPE[21]	250 mL HDPE[21]
Residue (Filterable)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Residue (Nonfilterable)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Residue (Settleable)	Cool to 4 °C.	N/A	48 hours	N/A	Imhoff Cone	N/A
Residue (Total)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Residue (Volatile)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Silica	Cool to 4 °C.	N/A	28 days	N/A	125 mL HDPE[21]	N/A
Specific Conductance	Cool to 4 °C.	N/A	28 days	N/A	250 mL HDPE[21]	N/A
Sulfate	Cool to 4 °C.	Cool to 4 °C.	28 days	28 days	125 mL HDPE[21]	125 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[21] HDPE - High-Density Polyethylene bottles.
[22] Metals and cyanide may be collected in the same container for soils.
[23] BR - Boston Round.

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Miscellaneous (Cont.)						
Sulfide	Cool to 4 °C, add 4 drops zinc acetate, and NaOH to pH > 9.	Add 2 N zinc acetate until moistened, and cool to 4 °C.	7 days	7 days	1 L HDPE[21]	250 mL CWM[11]
Sulfite	Cool to 4 °C.	N/A	ASAP	N/A	125 mL HDPE[21]	N/A
Surfactant	Cool to 4 °C.	N/A	48 hours	N/A	500 mL HDPE[21]	N/A
TCLP Extractable Fraction	Cool to 4 °C.	Cool to 4 °C.	14 days / NA / 14 days[24]	14 days / N/A / 14 days[24]	3 - 1 L AG[17]	500 mL CWM[11]
TCLP Volatile Fraction	Cool to 4 °C. No headspace.	Cool to 4 °C, No headspace.	14 days/7 days/ 40 days[24]	14 days/7 days/ 40 days[24]	500 mL glass PTFE-lined septa	125 mL glass with PTFE-lined septum or 25 g EnCore™ sampler
TCLP Inorganic Fraction (Mercury)	Cool to 4 °C.	Cool to 4 °C.	28 days / N/A / 28 days[24]	28 days / N/A / 28 days[24]	1 L HDPE[21]	500 mL CWM[11]
TCLP Inorganic Fraction (all other metals)	Cool to 4 °C.	Cool to 4 °C.	180 days / N/A / 180 days[24]	180 days / N/A/ 180 days[24]	1 L HDPE[21]	500 mL CWM[11]
Temperature	None required.	N/A	ASAP	N/A		
Total Organic Halogens (TOX)	Cool to 4 °C, H_2SO_4 to pH < 2.	Cool to 4 °C.	28 days	28 days	16 oz BR[23]	125 mL CWM[11]
Toxic Recoverable Petroleum Hydrocarbons	Cool to 4 °C, H_2SO_4 to pH < 2.	Cool to 4 °C.	28 days	28 days	2 - 1 L glass	250 mL CWM[11]
Turbidity	Cool to 4 °C.	N/A	48 hours	N/A	250 mL HDPE[21]	N/A

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[17] AG - Amber Glass bottle(s) with Teflon-lined cap(s).
[21] HDPE - High-Density Polyethylene bottles.
[23] BR - Boston Round.
[24] Holding times shown: from collection to toxicity characteristic leaching procedure (TCLP) extraction/from TCLP extraction to preparative procedure/from preparative procedure to analysis.

Appendix C
Environmental Sampling Instructions

C.1 Sampling Strategies

C.1.1 Scope and application. This instruction discusses strategies that can be employed to sample various media, including but not limited to soils, sediments, or water. Several different types of sampling strategies exist that can be categorized as either statistical or nonstatistical methods. Applications and limitations of each sampling strategy will be briefly described.

C.1.2 Sampling strategies. One of the main goals of any investigation is to collect samples that are representative of the site conditions so that an accurate assessment of contamination can be made with a minimum number of samples. Based on the conceptual site model (CSM) , crucial pathways and media requiring assessment are identified, and are later used to evaluate whether the data make sense for what is known about the site. The various sampling strategies available can be grouped into two basic categories: statistical and nonstatistical methods. To ensure that samples are as representative as possible, statistics are often used to design an appropriate sampling strategy and to provide a sound basis for supporting project decisions. Depending on data needed to support project decisions, input from an environmental statistician may be obtained. In addition, software programs (e.g., DQO Pro, DEFT, DataQuest, Visual Sampling Plan) are available to aid in the evaluation of various sampling scenarios and the uncertainty associated with them. Classical statistical methods are most applicable to sampling media that are considered fairly homogeneous (e.g., ground water, surface water). However, because of the spatial variability of soils, application of sampling strategies using classical statistical techniques may be limiting. The use of geostatistical methods is recommended for sediments and soils to account for the variability of the media. A related factor to consider is the distribution of the contaminant within the environmental medium, and how this may impact the use of the data or what is considered representative. Information on how the contaminant was dispersed at the time of waste generation, spill, or discharge may help in assessing whether the contamination is present on a molecular scale (e.g., solvent or solution spills) or on a macroscale (e.g., lead shot, ammunition debris, and TNT chunks). The latter situation increases the likelihood that samples may exhibit a high short-range heterogeneity, and the challenge of obtaining representative samples becomes even more difficult. The use of compositing and homogenizing techniques can improve representativeness of the samples (i.e., when amenable to the eventual physical/chemical analyses) by invoking the physical process of averaging. Refer to Instructions E-2 and E-3 of Appendix E for additional information on homogenizing and compositing techniques and to E-4 for information on the collection, handling, and storage of solid volatile organic analysis (VOA) samples. Statistics can also be used to determine the number of samples required to reach a prescribed level of uncertainty. However, when statistical calculations result in an unacceptably high number of samples being defined, the use of field analytical technologies or field screening techniques should be pursued to reduce the cost of sample analyses while maintaining a desired level of site coverage. Refer to Appendix H for additional information on this subject. Typically, more than one sampling strategy or approach is necessary when several media or types of contamination are under investigation, and most sampling plans employ a combination of sampling strategies. The following text and Table C-1 summarize basic descriptions, applications, and limitations for some frequently used sampling strategies. Additional references are also included to provide more detailed discussions on the subjects.

C.1.2.1 Classical statistical sampling. A discussion of statistical sampling is presented in the following sections. For a detailed discussion of classical statistical methods see U.S. Environmental Protection Agency (USEPA) EPA/530/SW-89/026, EPA/SW-846 (Volume II)), Gilbert (1987), and Pitard (1993).

Table C-1
Comparison of Sampling Strategies

Sampling Strategy	Description	Application	Limitations
		Classical Statistical Sampling Strategies	
Simple random sampling	Representative sampling locations are chosen using the theory of random chance probabilities.	Sites where background information is not available and no visible signs of contamination are present.	May not be cost-effective for samples located too close together. Does not take into account spatial variability of media.
Stratified random sampling	Site is divided into several sampling areas (strata) based on background or site survey information; each stratum is evaluated using a separate random sampling strategy.	Large sites characterized by a number of soil types, topographic features, past/present uses, or manufacturing/storage areas.	Often more cost-effective than random sampling. More difficult to implement in the field and analyze results. Does not take into account spatial variability of media.
Systematic grid sampling	Most common statistical strategy; involves collecting samples at predetermined, regular intervals within a grid pattern.	Best strategy for minimizing bias and providing complete site coverage. Can be used effectively at sites where no background information exists. Ensures that samples will not be taken too close together.	Does not take into account spatial variability of media.
Hot-spot sampling	Systematic grid sampling strategy tailored to search for hot spots.	Sites where background information or site survey data indicate that hot spots may exist.	Does not take into account spatial variability of media. Trade-offs between number of samples, chance of missing a hot-spot, and hot-spot size/shape must be weighed carefully.
Geostatistical approach	Representative sampling locations are chosen based on spatial variability of media. Resulting data are analyzed using kriging, which creates contour maps of the contaminant concentrations and the precision of concentration estimates.	More appropriate than other statistical sampling strategies because it takes into account spatial variability of media. Especially applicable to sites where presence of contamination is unknown.	Previous investigation data must be available and such data must be shown to have a spatial relationship.
		Nonstatistical Sampling Strategies	
Biased sampling	Sampling locations are chosen based on available information.	Sites with specific known contamination sources.	Contaminated areas can be overlooked if they are not indicated by background information or visual signs of contamination. Best if used with statistical approach, depending on the project objectives.
Judgmental sampling	An individual subjectively selects sampling locations that appear to be representative of average conditions.	Homogeneous, well-defined sites.	Not usually recommended due to bias imposed by individual, especially for final investigations.

C.1.2.1.1 Simple random sampling. Simple random sampling is the most basic statistical approach and is usually applied when minimal site background information (e.g., past practices, uses of hazardous materials, etc.) is available and visible signs of contamination are not evident during the initial site survey. This strategy uses the theory of random chance probabilities to choose representative sampling locations. Each sample location is chosen independently of any previously chosen sample location. It is most effective when the number of available sampling points is large enough to lend statistical validity to the random selection process. The simple random sampling approach may be more costly than other statistical methods since a larger number of samples may be required to characterize the site.

C.1.2.1.2 Stratified random sampling. Investigations of large sites that encompass a number of soil types, topographic features, or land uses may benefit by using a modified random sampling approach, called stratified random sampling. In this strategy, the site is divided into different sampling areas (strata) that are internally homogeneous based on existing data and background information. The division of the site into strata is based on the assumption that each stratum is more internally homogeneous than the site as a whole. Each stratum is sampled at locations based on a simple random sampling approach. By grouping similar sampling points and treating each group separately, each with its own individual random sampling scheme, the precision of the study is increased. In addition, this approach controls the variability due to contaminant concentration, location, terrain type. etc., and it often results in more efficient allocation of resources than would be possible with a simple random sampling method. Sampling analyses from each stratum may be used to determine the mean or total contaminant concentration within the stratum. However, data from each stratum may be used to make comparisons between the different strata or combined to provide information about the entire site.

C.1.2.1.3 Systematic grid. Systematic grid sampling, sometimes referred to as systematic random sampling, is the most common statistical sampling strategy. It involves collecting samples at predetermined. regular intervals (i.e., within a grid pattern). The location of the first sampling point is selected at random and all subsequent sample locations are determined using a systematic pattern from that point. This approach is typically used when a large site (e.g., measured in acres) must be sampled to characterize the presence and distribution of contaminants. The grid-based option is probably the best classical statistical sampling strategy for minimizing bias and providing complete site coverage. The most basic grid system is a straight line between two points on which regularly spaced sampling locations are designated. This one- dimensional sampling grid may be used for sampling along a straight drainage ditch or other man-made feature. However, most soil sampling schemes require a two-dimensional grid system for locating sampling points. The following types of grids are generally used: square, triangular, or other systematic pattern. Sampling is usually performed at each grid-line intersection. However, sampling in the center of each grid square/triangle or obtaining a composite of samples within a grid square/triangle is also acceptable.

C.1.2.1.4 Hot-spot sampling. "Hot spots" are usually defined as small, localized areas of a media that are characterized by high contaminant concentrations. In order to detect hot spots, a special systematic grid sampling approach is necessary. However, because all of the media cannot be sampled, there is still a possibility that hot spots exist even if none are discovered during the sampling process. Statistical approaches for detection of hot spots are discussed in Gilbert (1987). A hot-spot sampling plan should consider the following factors:

- Grid spacing and geometry. A triangular grid pattern increases the efficiency of the hot-spot search. In addition, the probability of finding hot-spots increases as the spacing between grid points decreases.

- Hot-spot shape/size. The larger a hot spot is, the more likely it is to be discovered. The shape of the hot spot also affects the probability of its being detected. Narrow or small-circular patterns may escape detection because they are located between grid sampling locations. Large-circular and wide-elliptical hot spots are the easiest to find.

- False negative rate. This measures the probability that a hot spot will be missed even if one is present.

C.1.2.1.5 Geostatistical approach. Classical statistical methods for the design of sampling projects are well-known and have been the standard approach in the past. However, these strategies have one major

drawback—they do not take the natural variability of the media into account. As such, they may not adequately characterize contamination at sites, especially those sites that are fairly heterogeneous and/or where the presence of contamination is unknown. Consequently, classical statistical methods are most appropriately applied to sites where the source of contamination is known (e.g., a landfill, waste pile, etc.) or small sites where the entire area is to be remediated as a unit (e.g., in the case of soils, the entire site will be solidified). To more accurately characterize sites where the presence of contamination is unknown, statisticians now believe that geostatistical methods are more appropriate than classical statistical methods because they take into account the spatial variability of the media when estimating contaminant concentrations. Geostatistical methods may be used for sampling naturally occurring materials such as soils or ground water and man-made units such as landfills or waste piles. Characterization of any media is difficult because contaminant levels are spatially correlated. This means that contaminant concentrations from samples taken close together are more likely to be similar than contaminant concentrations from samples taken farther apart (regional variability). Geostatistics describes how to sample and analyze regional variability by defining the representativeness of a sample in terms of its range of correlation or zone of influence. For example, a sample location selected through geostatistics will represent a circular area with a radius less than or equal to the zone of influence. In other words, the sample would be representative of the media within the circular area. A two- stage sampling approach is typically used in geostatistical sampling strategies. Initially, a sampling survey is performed to collect basic data. These data are used to create a graph that defines the distance over which samples are representative. This graph is then used to dictate the shape, size, and orientation of another systematic grid that is used in the second, final sampling event. Geostatistical sampling strategies are relatively complex, and further discussions of this approach are found in Engineer Technical Letter (ETL) 1110-1-175, Borgman and Quimby (1988), and Gilbert (1987).

C.1.2.2 Nonstatistical sampling. Types of nonstatistical sampling are described as follows.

C.1.2.2.1 Biased sampling. Biased sampling is used to evaluate sites with specific, known sources of contamination (e.g., the site survey discovered visible signs of contamination or records indicate that certain locations are suspect based on past/present practices). As such, sampling locations are chosen based solely on available information.

C.1.2.2.2 Judgmental sampling. In judgmental sampling schemes, an individual subjectively selects the sampling locations that appear to be representative of average conditions. If the individual is knowledgeable, judgmental sampling can result in accurate estimates of site conditions. Although a certain amount of judgment is necessary in any sampling approach, total reliance on judgment decisions is not recommended because an individual's bias often leads to poor quality data and improper conclusions. However, if judgmental sampling is necessary, multiple samples should be collected to add some measure of precision.

C.1.3 Potential problem. Table C-1 shows the limitations associated with these sampling strategies.

C.2 Ground Water Sampling

C.2.1 Scope and application. This instruction presents guidelines for collecting representative ground water samples from temporary and permanent ground water monitoring wells and, where applicable, from other direct push well screen samplers. Guidance for the installation of temporary wellpoints by direct push methods for sampling ground water at discrete points may be found in American Society for Testing and Materials (ASTM) D 6001. Typical ground water monitoring wells are 5 or 10 cm (2 or 4 in.) in diameter and are constructed of polyvinyl chloride (PVC) or stainless steel. Instructions presented herein are intended to include sample collection from wells that have not been completed as production or extraction wells. The instructions can be used to identify an appropriate sampling protocol for the acquisition of a representative sample. Instruction C-4, "Potable Water Sampling," includes procedures for sampling permanent production wells or any other well constructed with a discharge tap. Instructions for purging and sampling wells by the following techniques are included in this section: bailer, portable submersible pump, bladder pump, hand pump, centrifugal pump, peristaltic pump, air lift pump, and low-flow sampling.

C.2.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs identified in the hazardous, toxic, and radioactive waste (HTRW) technical planning process. The sampling strategy developed for a particular site will influence several project decisions, including, but not limited to, sampling locations, types of samples, sampling frequency, and sampling and analytical protocols. Sampling strategies may be significantly influenced by such factors as physical site constraints, safety, and cost, to name a few. The technical planning process that results in the development of the sampling strategy is critical because of the difficulty in acquiring representative samples, the reduction of contaminant action levels, and the problems associated with trace level cross-contamination. Successful investigations of hazardous waste sites are highly dependent on an effective sampling scheme. Development of a sampling scheme for purposes of characterizing a hazardous waste site should follow the fundamentals of the scientific approach. A successful sampling scheme requires a logical design to allow an evaluation of potential contaminants in relation to background conditions, vertical extent, horizontal extent, and mobility in various media. Additional guidance may be found in ASTM D 5903.

C.2.2.1 Sampling locations. Sampling at hazardous waste sites is usually conducted in an attempt to identify contamination and to define its extent and variability. With such an objective, it is most logical to choose sample locations that will yield the most information about site conditions. During evaluation of a site, sampling can be conducted by random, systematic, or biased sampling. Instruction C-1 discusses random, systematic, and biased sampling in detail. Often biased and random sampling techniques can be used together to thoroughly address an entire site. Some wells may be biased to potentially contaminated areas (e.g., former wastewater lagoons, former process or disposal areas), or potentially impacted areas (e.g., down-gradient locations). In areas less likely to be contaminated or areas with little available background information, random samples may be used to allow adequate assessment of the entire site. Ground water monitoring wells are positioned at locations and depths to satisfy ground water monitoring objectives. Ground water samples collected from monitoring wells are evaluated as discrete samples collected from the same location. Ground water samples collected from the same well are distinguished from one another because they are distributed through time. Unless each ground water monitoring well has a sampler dedicated to the well, the order of sampling monitoring should be from the least contaminated to the most contaminated.

C.2.2.2 Sample type. Ground water samples are typically discrete samples. A discrete (grab) sample is a discrete aliquot representative of a specific location at a given point in time. The sample is collected at once and at a particular point in the sample matrix. The representativeness of such samples is defined by the nature of the materials being sampled. In general, since contamination in ground water disperses over time and distance, it will take more grab samples to characterize the extent of contamination as the time from a release increases.

C.2.2.3 Suggested samplers. Each sampling technique presents various disadvantages and advantages for its application. For example, sample disturbance, sample volume, chemical/physical reactivity between potential contaminants and sampling tool materials, well diameter, depth to ground water, limitations of lift capacity of the sampling device, specified analytical parameters, and ease of decontamination vary from technique to technique. The advantages and disadvantages of each sampling technique are presented in the discussion of the technique.

C.2.2.4 Sample frequency. Contaminant concentrations in ground water vary across both time and space. Therefore, it is important to consider the potential temporal variability of the data collected. Determination of the number of samples needed to characterize a site is dependent upon the objectives and the site-specific conditions. For example, if the objective of the event is to determine whether the site is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, the site is contaminated and the objective is to establish the boundaries of the ground water contamination or trends in the data over time, a greater number of samples may be needed. Often statistical considerations can be helpful in determining sampling strategy.

C.2.3 Sample preservation and handling. Many chemical constituents and physicochemical parameters that are to be measured or evaluated in ground water monitoring programs are not chemically stable, and therefore sample preservation is required. Appropriate preservation techniques for various parameters and sample containers that should be used for each constituent or common set of parameters are specified in Appendix B. These preservation methods and sample containers are based on "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods" (EPA/SW-846). Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2 of Appendix F, "Packaging and Shipping Procedures." Improper sample handling may alter the analytical results of the sample, causing the results to be invalid. Samples should be transferred in the field from the sampling equipment directly into the container required for that analysis or set of compatible parameters. The sample should then be preserved in the field as specified in Appendix B. Because of the low analytical detection limits required for certain data uses, care must be taken when collecting the sample to avoid the loss/gain of any contaminants. The samples for volatile analysis should be carefully transferred directly from the sample collection device to the sample container to minimize contaminant loss through agitation/volatilization or adherence to another container. Samples should be collected in the order of the parameters listed in Section C.2.3.1. If more than one container is required per parameter, the sample should be split equally among all containers until filled. Containers used to collect samples for organic analyses should not be prerinsed with water because of the possibility of preservation loss or the loss/gain of contaminants that may taint the analytical results. Decisions to filter samples are dictated by sampling objectives. Consideration should be given to what the application of field filtration is trying to accomplish. 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-line filtration is recommended because it provides better consistency through less sample handling and minimizes sample exposure to the atmosphere. Filters must be prerinsed following manufacturers' recommendations or using a minimum of 1 L of ground water following purging and before sampling. Further information on filtration considerations and consequences is given in Instruction E-1, Appendix E.

C.2.3.1 Sample containers. When metals are the analytes of interest, high-density polyethylene containers with polytetrafluoroethylene-lined polypropylene caps should be used. (Polytetrafluoroethylene is commonly referred to using the registered name of Teflon. Polytetrafluoroethylene will be referred to as PTFE.) When organics are the analytes of interest, glass bottles with PTFE-lined caps should be used. Refer to Appendix B or the specific analytical method for acceptable containers. Containers should be cleaned based on the analyte of interest. Instruction E-6, "Decontamination Procedures" (Appendix E), contains

additional information on appropriate glassware cleaning protocols. If precleaned bottles are used, the cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Refer to Appendix B for information on the required size and type of sample containers. Samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. The following is a preferred collection order for some common ground water parameters:

- Volatile organics (VOA)
- Purgeable organic carbon (POC)
- Purgeable organic halogens (POX)
- Total organic halogens (TOX)
- Total organic carbon (TOC)
- Extractable organics
- Total metals
- Dissolved metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionuclides

C.2.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action, retard hydrolysis, and reduce sorption effects. Preservation methods are generally limited to pH control, chemical addition, cooling, and protection from light. Prepreserved sample containers are not recommended. Because different amounts of preservative may be necessary to bring the sample to the required pH, it is recommended to add the preservative to the container in the field and to verify that the pH of the sample has been achieved. This information should be documented within field logbooks. The sampler should refer to Appendix B or the specific preservation method in EPA/SW-846 for the appropriate preservation technique.

C.2.3.3 Special handling for VOA samples. Water samples to be analyzed for purgeable organic compounds should be stored in 40-mL vials with septum inserts and screw caps. The septum should be placed on the sample vial so that the PTFE side is in contact with the sample. The 40-mL vials should be completely filled to prevent volatilization. Extreme caution should be exercised when filling a vial to avoid any turbulence that could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to pour the last few drops into the vial gently so that surface tension holds the water in a convex meniscus. The septum is then applied and some overflow is lost, but air space in the bottle is eliminated. After the bottle is capped, it should be turned over and tapped to

check for bubbles. If any bubbles are present, the procedure must be repeated. Care should be taken to ensure that no loss of preservative occurs, if applicable.

C.2.3.4 Special precautions for trace contaminant sampling. Contaminants can be detected in the parts per billion and/or parts per trillion ranges. Therefore, extreme care must be taken to prevent cross-contamination of these samples. The following general precautions should be taken when sampling:

- A clean pair of new disposable gloves should be worn each time a different location is sampled, and gloves should be donned immediately before sampling.
- To prevent cross-contamination between samples, it is suggested that the multiple VOA vials from each sampling location be sealed in a separate smaller plastic bag when the sampled medium is suspected of containing high concentrations of volatile organics.
- Sample containers filled with source or waste samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting and preserving, and activated carbon should be included in the bags to prevent cross-contamination.
- If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants should be lined with new, clean plastic bags.
- If possible, one member of the field team should take all the notes, fill out sample tags, field sheets, etc., while the other members collect all of the samples.
- Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.
- Field personnel should use equipment constructed of PTFE, stainless steel, or glass that has been properly precleaned. PTFE or glass is preferred for collecting samples where trace metals are of concern.
- Adequate field control samples should be collected.

C.2.4 Sampling methods. Sampling instructions for the most common techniques for collecting ground water samples from ground water monitoring wells are presented in this section. A summary of the methods is presented in Table C-2. Additional information is presented in EPA/540/S-95/504, EPA/625/R-93/003a, ASTM D 4448, ASTM D 6232, and ASTM D 6452. Additional information on the installation, development, and decommissioning of monitoring wells can be found in ASTM Standards D 5092, D 5299, and D 5521. After installation, the well should be developed to remove any fine material adjacent to the well casing. Wells should not be sampled immediately after development, due to the time needed to reach chemical equilibrium with the well construction materials. This lag time may often extend beyond 1 week. Consequently, well development is not addressed in this sampling and analysis guidance document. Refer to EM 1110-1-4000 for further information on well development. Once a well has been

Table C-2
Summary of Ground Water Sampling Techniques

	Advantages				Disadvantages			
Method	Compatible Construction Material	Ease of Use	Economical	External Power Source Needed	VOC Degassing	Ease of Decontamination	Large Water Volumes	Restrictive Ground Water Depth
Bailer	•	•	•			•		
Submersible Pump	•			•	•		•	
Bladder Pump	•			•				
Hand Pump	•	•	•		•			
Centrifugal Pump			•	•	•		•	•
Peristaltic Pump	•			•	•	•		•
Air-Lift Pump		•		•	•		•	•
Passive Samplers	•	•	•					

located and properly identified, field measurements should be noted in a bound field logbook. A cross-reference should be made between the previously recorded physical location/identification locating the well to be sampled to ensure the proper well has been selected. Misidentification of a sampling point in the field will result in erroneous data that may affect management decisions. Also included in field measurements are the physical measurements of the well and its physicochemical parameters. Physical measurements that may be recorded in the field logbook include the presence and diameter of protective casing, diameter and construction material of the well casing, total depth of well from the top of casing, surveyors' mark, depth from top of casing to water, and the volume of water in the well and filter pack. The volume of water can be calculated by the submerged length of the well and calculating the volume of water in the submerged casing and filter pack. The total depth of the well should not be measured before sampling, but be obtained from well logs. Measuring to the bottom of the well casing may cause resuspension of settled solids from the formation materials and require longer purging times for turbidity equilibration. The well depth should be measured after sampling is complete. Volumes of water in various well casing diameters are listed in Table C-3.

Table C-3
Water Volume in Casing

Nominal Casing Diameter centimeters (inches)	Water Volume liters/linear meter (gallons/linear foot)
5.1 (2)	2.03 (0.16)
10.2 (4)	8.11 (0.65)
15.2 (6)	18.24 (1.47)
20.3 (8)	32.43 (2.61)
25.4 (10)	50.67 (4.08)
30.5 (12)	72.96 (5.88)

The volume of water in the filter pack should be calculated assuming a porosity of 30 percent within the filter pack. The volume of water present in the well casing and filter pack may be calculated as shown in the following example:

Assumptions: 2-in. well casing; well depth is 100 ft below ground surface, ground water depth is 20 ft below the ground surface, and the boring diameter is 8 in.

Volume of water in well = (well depth - depth to water) × (water volume in casing)
= (100 - 20) ft (0.1632 gal/ft)
= 13.0 gal

Volume of water in filter pack = (volume of filter pack) × 30%/231 $in.^3$/gal
= {[(•) (8 in.)²/2] × 80 ft (12 in./ft)} × 0.3/231 $in.^3$/gal
= 58.7 gal

Total volume in well casing and filter pack = 13 gal + 58.7 gal
= 71.7 gal

The volume of water in any size casing can be determined using the following formula:

No. of gallons = $5.8752 \times C^2 \times H$

where C = casing diameter, ft

H = height of water column, ft

Besides the physical measurements taken as described previously and other information that may identify the well, physicochemical information such as dissolved oxygen, pH, specific conductance, temperature, and turbidity should be recorded initially (and in that order), during purging and before sampling (see Section C.2.4.1).

C.2.4.1 Well purging. To obtain a representative sample of ground water from a ground water monitoring well, the water that has stagnated and/or thermally stratified in the well casing must be purged or evacuated. The purging procedure allows fresh or representative ground water to enter the well. The optimum or preferred method to ensure that fresh water representative of the aquifer in contact with the well screen is being sampled is to perform a controlled sampling experiment. When indicator parameters such as specific conductance, pH, temperature, turbidity, and dissolved oxygen are stabilized, the well is presumed to be adequately flushed for the representative sample. In some instances, purging rates must be kept below 500 mL/min to avoid overpumping or pumping the well to dryness. Guidance on recommended stabilization criteria for the indicator parameters is included within the individual sampling equipment instructions. Ideally, wells should never be pumped to dryness. To accomplish this, pump rates may be adjusted, sometimes to less than 500 mL/min, and pumping times extended. Pumping with low-flow rates may also reduce the need for filtering water samples. Additional guidance on low-flow purging may be found in EPA/540/S-95/504. The volume of purging is significantly less in low-flow sampling, because the flow rate is designed to be low enough that flow is being induced only horizontally through the screen at a rate that replaces the evacuated water. This flow rate (typically 100-500 mL/min) will also have minimal effect on entraining particulates, which lowers the sample turbidity, which is crucial to metals analyses. The well can be purged or evacuated in several ways. In any instance it is paramount to ensure that the purging procedure does not cause cross-contamination from one well to the next. Therefore, the preferred method

employs dedicated equipment and pumps. Because, commonly, it may not be practicable to dedicate a pump to a specific well, it is permissible to decontaminate this equipment, using approved methods. Tubing should always be dedicated and never used for more than one well. The selection of an evacuation method most often relies on the depth to water (DTW) in the well. If the static DTW is less than 7.6 m (25 ft), a hand pump or bailer may be the best method for evacuation. If the static DTW is greater than 7.6 m (25 ft), a submersible pump should be used. As mentioned earlier, care must be taken to ensure that this does not act as a route of cross-contamination. Pumps should be decontaminated between well locations. During evacuation, pump intake must not be set greater than 1.8 m (6 ft) below the dynamic water level. This requires that the evacuation device be lowered as purging continues and the water level drops. Hand bailing may be utilized with a static water level greater than 7.6 m (25 ft) if a submersible pump is not available or with a static water level less than 7.6 m (25 ft) if other conventional pumps are not available. However, use of a bailer is not recommended because it has the potential to aerate the well water, produce inadequate removal of fines, concentrate floating product on the bailer that may introduce contamination, introduce contaminants from inside the well casing, and cause nonsteady removal of water, which may result in dilution instead of evacuation of the well. In general, the mechanics of the hand-bailing method for well purging may introduce contamination potential and variability. There are many pumps that may be used for well purging. Not all pumps are acceptable under all conditions. The preferred and most commonly used pumps are centrifugal and peristaltic pumps (when depth to water is less than 7.6 m (25 ft)) and submersible pumps (when depth to water is greater than 7.6 m (25 ft)). Information on various pumps and methods of purging is provided later in this instruction. Recent studies have found that some in situ ground water sampling devices minimize or eliminate the need for purging (EPA/600/S4-90/028). These devices are stand-alone systems installed directly into the subsurface and are not used to sample existing ground water monitoring wells. For existing ground water monitoring wells, innovative samplers have been developed that sample the ground water monitoring well without the need to purge the well initially. These diffusion samplers have shown through case studies to be an effective means of acquiring volatile organic compound (VOC) samples (Vroblesky and Hyde 1997). Occasionally, a light, nonaqueous phase layer (LNAPL) (floating product) may be present in a monitoring well designated for sampling. If it is suspected that the well contains an LNAPL, an interface probe should be used to verify its presence. If an LNAPL is present, the thickness should be measured and an appropriate inert bailer should be used to collect a sample of the floating product. Whenever possible, measurements of the free product should be taken using either steel tape and paste or an interface probe. A bailer can significantly under- or overestimate the thickness of free product in the well and should not be used for determining the elevations of air/free product and free product/water interfaces. The use of bailers should be limited to verification of the presence of free product in a well or collection of a sample of it (EPA/510/R-96/001). Collection of a ground water sample may not be appropriate if an LNAPL is present in the well, for the sampler will likely become contaminated as it passes through the LNAPL to reach and sample the ground water below.

C.2.4.2 Bailers. Bailers are one of the simplest and most commonly used sampling methods for sampling ground water monitoring wells (Figure C-1).

C.2.4.2.1 Applicability. Bailers are constructed of a variety of materials compatible with the parameter of interest. They are economical and convenient enough that a separate bailer may be dedicated to each well to minimize cross-contamination. An external power source is not required. Bailers provide a low surface-to-volume ratio, which reduces degassing of VOCs. Cross-contamination can be a problem if the bailer is not adequately decontaminated. Bailers offer a relatively limited sample volume and may not be desirable for purging a well if large amounts of water need to be removed from the well for purging. The sampling technique used with bailers may, however, cause a surging action on the well, which may increase the turbidity of the well sample. Consequently, bailers have a higher potential for loss of volatiles and are not recommended for either volatile or metals sampling. Where representative numbers for metals and

volatiles are required, it is recommended that the low-flow procedures detailed in Section C.2.4.9 be utilized.

C.2.4.2.2 Method summary and equipment. Bailers are manufactured in numerous types, sizes, and construction materials. Bailers are typically weighted lengths of pipe attached to a cord with a check valve at the lower end. They are typically constructed of PVC, PTFE, or stainless steel. The PTFE bailer is recommended if the bailer is used to collect ground water samples for VOC analysis. Bailers can be dedicated to a specific well, i.e., used only for purging and sampling that well. Dedicated bailers are typically stored to prevent cross-contamination or, less preferably, left hanging in the well itself. It should be noted that stainless steel bailers left stored in a well will likely rust under high-humidity conditions. The bailer should be decontaminated after each use. Disposable bailers are also available and are cost-effective for certain investigations. Haul lines for bailers may consist of PTFE-coated stainless steel cable, polyethylene rope, or nylon rope. Of these three, nylon rope is the least desirable because it may introduce contaminants. The use of braided rope is discouraged, because it cannot be decontaminated. For each sampling event, the rope for dedicated bailers should be changed following purging and before sampling. For nondedicated bailers, rope should be changed between wells. After removal, the rope should be properly discarded.

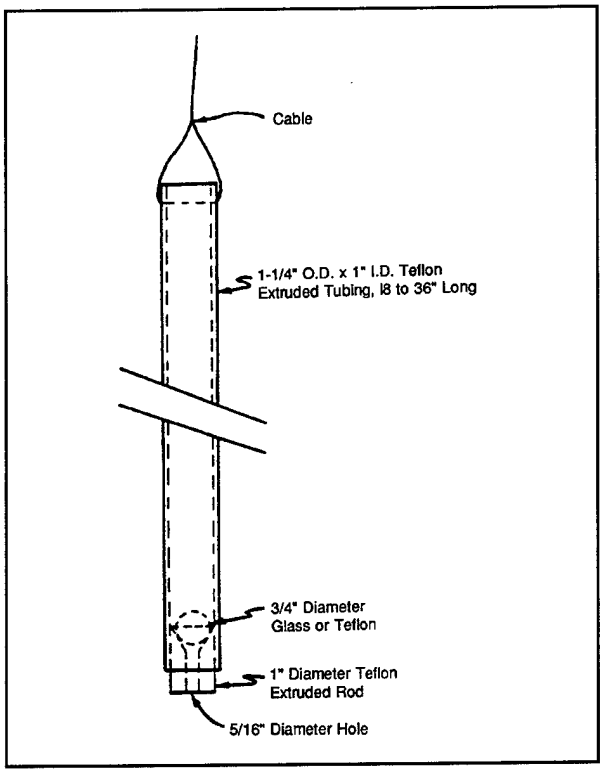


Figure C-1. Bailer (to convert dimensions to mm, multiply by 25.4)

C.2.4.2.3 Sampling procedure.

- Prepare the work area outside the well by placing plastic sheeting on the ground to avoid cross-contamination.
- Determine the saturated water column in the well using an electronic water level indicator. Calculate the fluid volume in the casing and determine the amount of water to be removed for purging.
- Attach a decontaminated bailer to cable or line for lowering, or use dedicated bailer already in well.
- Lower bailer slowly until it contacts water surface.
- Allow bailer to sink and fill with a minimum of surface disturbance.
- Slowly raise bailer to surface. Do not allow bailer line or bailer to contact ground.

- Purge well until the dissolved oxygen (DO), pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)

- Ensure that equilibrium is established by making three consecutive readings, where one casing volume is pumped between each reading.

- Collect VOA samples first following guidelines specified within this appendix.

- Fill sample bottles for remaining parameters by tipping bailer to allow slow discharge from top to flow gently down the side of the sample bottle with minimum entry turbulence. If a bottom drain is present on the bailer, recommended that a slow steady flow be achieved.

- Repeat these steps as needed to acquire sufficient volume to fill all sample containers.

- 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.

- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.

- Record the information in the field logbook and field sheet, and complete all chain-of-custody documents (see Instruction F-1, "Documentation," in Appendix F).

- Thoroughly decontaminate the bailer after each use, regardless of whether the bailer is dedicated to one well or used to sample other wells.

- Close well.

C.2.4.3 Portable submersible pump. Portable submersible pumps are an effective technique for pumping large volumes of water at a steady rate but require an external electrical power source.

C.2.4.3.1 Applicability. Advantages of submersible pumps include their ability to pump variable amounts from various depths. This advantage makes these pumps applicable not only for purging and sampling but also for aquifer characterization tests. Pumping rates for various units range from as little as 100 mL per minute to 3,784 L per minute (1,000 gpm). The pumping rate for most units can be individually adjusted. Disadvantages of submersible pumps are that they require an external electrical power source and may be difficult to decontaminate between wells. The propeller construction of the pump assembly may cause degassing of volatile organic compounds; therefore, some states or USEPA regions may restrict the use of submersible pumps when sampling for VOCs.

C.2.4.3.2 Method summary and equipment. For submersible pumps, the pump assembly, the electric drive motor, and associated hoses and electrical cables are suspended from a steel cable or discharge pipe and submerged in the well. Intake is typically located between the motor and the pump assembly.

Horsepower, head, and lift capacity range widely. Submersible pumps are available for 5-cm (2-in.) and larger wells. Some pumps are constructed of stainless steel and PTFE to maintain sample integrity. Submersible pumps far exceed the pumping limitations of other sampling equipment.

C.2.4.3.3 Sampling procedure. Recommended sampling procedures are as follows:

- Prepare the work area outside the well by placing plastic sheeting on the ground to avoid cross-contamination.
- Determine the saturated water column in the well using an electronic water level indicator. Calculate fluid volume in the casing and determine the amount of water to be removed for purging.
- Lower the decontaminated pump to below the water level and begin pumping. Collect or dispose of purged water in an acceptable manner. Lower the pump as required to maintain submergence.
- Measure rate of discharge frequently. A bucket and stopwatch are commonly used.
- Purge well until the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)
- Ensure that equilibrium is established by making three consecutive readings, where one casing volume is pumped between readings.
- Reduce the pump discharge rate to less than 500 mL/min. Collect VOA samples first following guidelines specified within this appendix.
- Fill bottles for remaining parameters by allowing pump discharge to flow gently down the side of the bottle with minimal entry turbulence. Cap each bottle as filled.
- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.
- Ensure that the PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.
- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.
- Complete chain-of-custody documents, field logbook, and field sheet (see Instruction F-1, "Documentation," in Appendix F).
- Pull pump and allow system to drain and decontaminate.
- Close well.

C.2.4.4 Bladder pump. Bladder pumps employ a closed collection system that eliminates agitation and air or gas contact with the sample (Figure C-2).

C.2.4.4.1 Applicability. Advantages of the bladder pump include its ideal design for sampling wells for VOC analysis from wells as small as 5 cm (2 in.) in diameter. The pump can pump water from various depths and at adjustable rates. It can operate in low-yielding wells without the possibility of burning out the pump if the well is pumped dry. The inlet for the pump body is typically at the lower end, thus requiring minimum submergence. Top-ended inlet pumps are also available for floating product recovery. Disadvantages of the bladder pump include its relatively low pumping rate. It also requires an outside power source of compressed air or gas and may be difficult to decontaminate between wells.

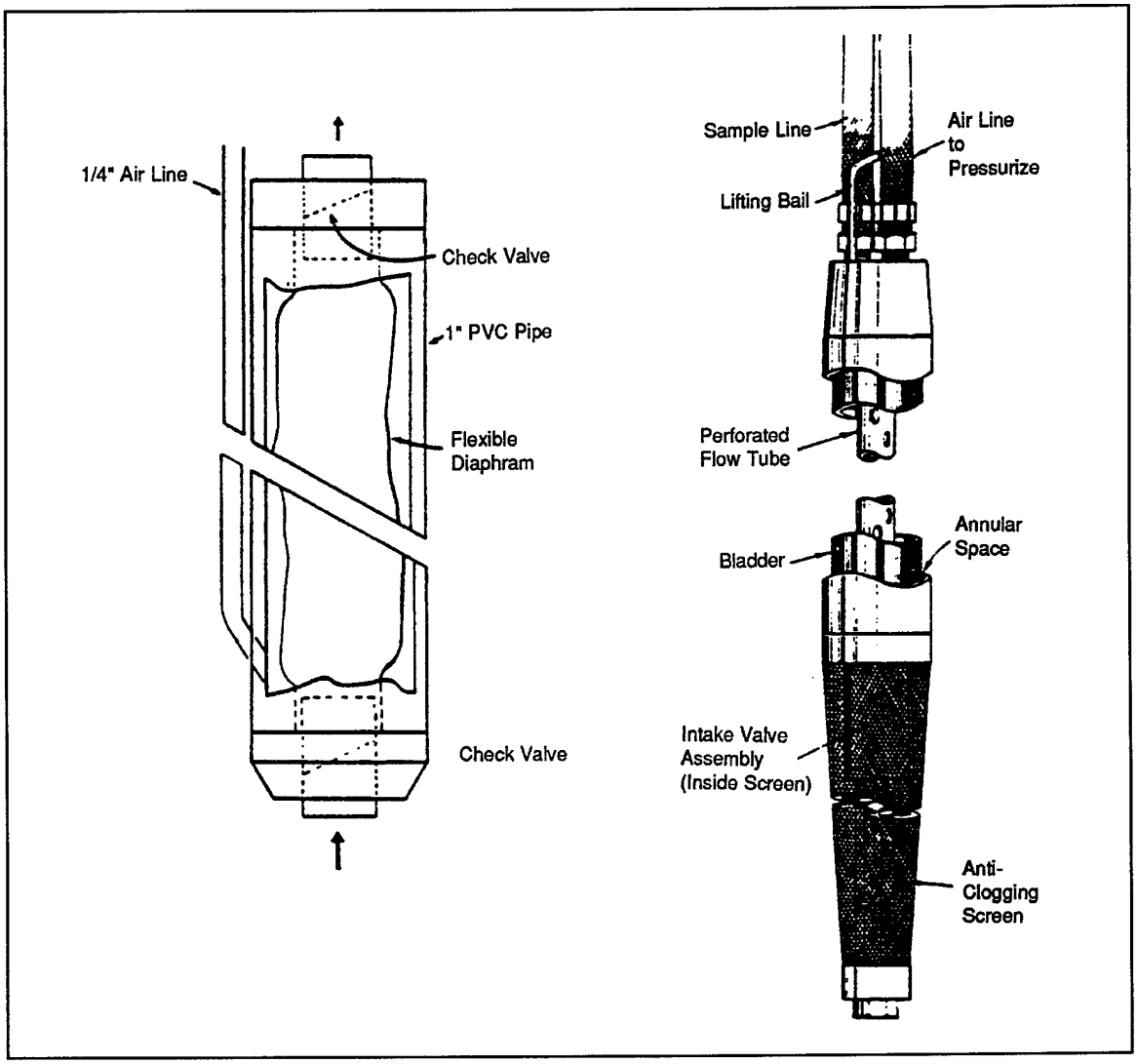


Figure C-2. Bladder pump (to convert dimensions to mm, multiply by 25.4)

C.2.4.4.2 Method summary and equipment. The closed system provides the best method available for sampling wells for VOCs. The pump fills through a lower check valve under hydrostatic pressure, collapsing the bladder in the pump body. The bladder is then pressurized with gas or air causing it to expand, thus applying pressure in the pump body. The bladder is pressurized using a control box and air compressor assembly. This in turn closes the lower check valve and forces the contents of the pump body up through the sample line check valve. Venting the bladder will allow the pump to refill and begin another cycle. An inflatable packer is often used in conjunction with bladder pumps to reduce the amount of water to be purged for sampling. The packer is often positioned immediately above the well screen so that only water in the screened area of the well will require purging once the packer is properly inflated.

C.2.4.4.3 Sampling procedure. Recommended sampling procedures are as follows:

- Prepare the work area outside the well by placing plastic sheeting on the ground to avoid cross-contamination.

- Determine the amount of water to be removed for purging. Determine the saturated water column in the well using an electronic water level indicator. Calculate the fluid volume in the casing if an inflatable packer is not present in the well. If an inflatable packer is present in the well, refer to construction diagrams of the well to determine the saturated water column below the packer. Make sure that the packer is not within the screened interval.

- Attach a pressurized air hose to the packer connection (if present) and inflate packer to proper pressurization level, typically 60 to 80 psi. After the packer is inflated, reattach pressurized air hose to the bladder pump connection and purge the well as discussed in Section C.2.4.4.2.

- Measure the rate of discharge frequently. A bucket of known volume and a stopwatch are commonly used.

- Purge well until the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)

- Ensure that equilibrium is established by making three consecutive readings, where one casing volume is pumped between each reading.

- Collect VOA samples first following guidelines specified within this appendix.

- Fill sample bottles of remaining parameters by allowing pump discharge to flow gently down the side of the bottle with minimal entry turbulence. The pump discharge rate should be less than 500 mL/min. Cap each bottle as filled.

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

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

- Label the sample with an appropriate label. Be sure to complete the label with all necessary information.

- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.

- Complete chain-of-custody documents, field logbook, and appropriate field sheet (see Instruction F-1, "Documentation," in Appendix F).

- Depressurize packer (if present), remove the pump, and close well.

C.2.4.5 Hand pumps. Hand pumps are positive displacement pumping systems designed for developing, purging, and sampling (for some analyses) 5-cm (2-in.) or larger ground water monitoring wells.

C.2.4.5.1 Applicability. Hand pumps do not require an external power source. Units are easily transported. A sustained pumping rate can be achieved. The hand pump could cause cross- contamination if the unit is not thoroughly decontaminated. The hand pump may not be suitable for collecting samples for VOCs because the pump creates a vacuum pressure on the water during operation, which may result in degassing of volatile compounds.

C.2.4.5.2 Method summary and equipment. Hand pumps are readily transportable and can be used to provide sampling in remote areas. Hand operation enables the user to vary the pumping rate to more than 4 gpm at depths exceeding 30 m (100 ft). Hand pumps are typically constructed of offset sizes of PVC piping and check valves. Typically, a small-diameter pipe resides within a larger diameter pipe. The small-diameter pipe is forced up and down and the resulting movement creates a positive displacement.

C.2.4.5.3 Sampling procedure. Recommended sampling procedures are as follows:

- Prepare the work area outside the well by placing plastic sheeting on the ground to prevent cross-contamination.

- Determine the saturated water column in the well using an electronic water level indicator. Calculate the fluid volume in the casing and determine the amount of water to be removed for purging..

- Lower the decontaminated hand pump assembly into the well and begin operating the pump in a steady motion.

- Measure the rate of discharge frequently. A bucket of known volume and a stopwatch are commonly used.

- Purge well until the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)

- Ensure that equilibrium is established by making three consecutive readings, where one casing volume is pumped between each reading.

- Collect VOA samples first.

- Fill sample bottles for remaining parameters by allowing pump discharge to flow gently down the side of the bottle with minimal entry turbulence. The pump discharge rate should be less than 500 mL/min. Cap each bottle as filled.

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

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

- Label the sample with an appropriate label. Be sure to complete the label with all necessary information.

- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.

- Complete chain-of-custody documents, field logbook, and appropriate field sheet (see Instruction F-1, "Documentation," in Appendix F).

- Remove the pump assembly and decontaminate.

- Close the well.

C.2.4.6 Centrifugal pump. A centrifugal pump is a type of suction pump used to purge wells.

C.2.4.6.1 Applicability. Advantages of centrifugal pumps include their ability to provide substantial pumping rates and their ready availability. Disadvantages are that they require an external power source and may be difficult to decontaminate between wells. The materials with which these pumps are constructed may frequently be incompatible with certain sample analytes. The centrifugal pump is not suitable for collecting samples for VOC analysis because the pump creates a vacuum pressure on the water during operation, which results in degassing of volatile compounds. These pumps cannot pull water more than 6 m (20 vertical ft).

C.2.4.6.2 Method summary and equipment. Centrifugal pumps are a type of suction pump. An impeller rotating inside the pump chamber discharges water by centrifugal force. The resulting pressure drop in the chamber creates a suction that causes water to enter the intake pipe in the well. Since entrance of water into the intake depends on atmospheric pressure, the height of the intake lift is limited to about 6 m (20 ft) at sea level and less at higher altitudes. Discharge rates from 19 to 151 L per minute (5 to 40 gpm) can be attained and water can be pushed as high as 46 m (150 ft) above the pump. Pumps are typically motorized by a small gasoline engine attached to the pump.

C.2.4.6.3 Sampling procedure. Recommended sampling procedures are as follows:

- Prepare the work area outside the well by placing plastic sheeting on the ground to avoid cross-contamination.

- Determine the saturated water column in the well using an electronic water level indicator. Calculate the fluid volume in the casing and determine the amount of water to be removed for purging.

- Lower decontaminated intake hose into well.

- Prime pump with distilled water and start pump.

- Containerize or discharge purge water accordingly.
- Measure the rate of discharge frequently. A bucket of known volume and a stopwatch are commonly used.
- Purge well until the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)
- Ensure that equilibrium is established by making three consecutive readings, where one casing volume is pumped between each reading.
- Collect VOA samples first.
- Fill sample bottles for remaining parameters by allowing pump discharge to flow gently down the side of the bottle with minimal entry turbulence. The pump discharge rate should be less than 500 mL/min. Cap each bottle as filled.
- Preserve the samples as necessary and verify that the pH is sufficient for the criteria.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.
- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.
- Complete chain-of-custody documents, field sheet, and field logbook (see Instruction F-1, "Documentation," in Appendix F).
- Close the well.

C.2.4.7 Peristaltic pump. Peristaltic pumps operate in a manner similar to that of centrifugal pumps but displace the fluid by mechanical peristalsis (Figure C-3).

C.2.4.7.1 Applicability. An advantage of the peristaltic pump is its design, which isolates the sample from the moving part of the pump and allows for easy decontamination by removal or replacement of the flexible tubing. Tubing can be dedicated to wells to reduce decontamination time. Disadvantages of these pumps include their low pumping rates and their limited height of intake lift (less than 6 m (20 ft)). These pumps also require an outside power source and, like other suction pumps, are not suitable for collecting samples for VOC analysis because of potential degassing effects.

C.2.4.7.2 Method summary and equipment. A flexible sampling tube is mounted around the pump chamber, and rotating rollers compress the tubing, forcing fluid movement ahead (the peristaltic effect) and

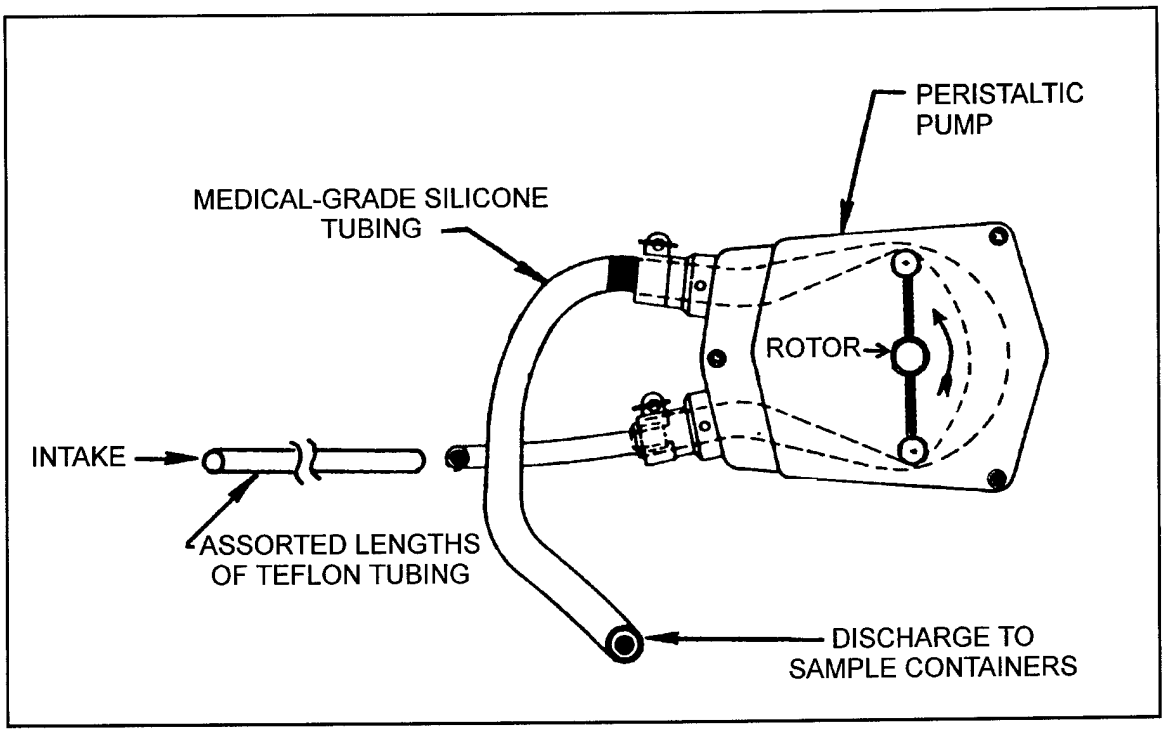


Figure C-3. Peristaltic pump

inducing suction behind each roller (Figure C-3). Peristaltic pumps generally have very low pumping rates suitable only for sampling shallow water tables in small-diameter wells.

C.2.4.7.3 Sampling procedures. Recommended sampling procedures are as follows:

- Prepare the work area outside the well by placing plastic sheeting on the ground to avoid cross-contamination.
- Determine the saturated water column in the well using an electronic water level indicator. Calculate the fluid volume in the casing and determine the amount of water to be removed for purging.
- Install clean medical-grade silicone tubing in the peristaltic pump head and attach the silicone tubing to the glass tubing outlet from the sample bottle.
- Attach the inlet glass tubing from the sample bottle to the required length of new PTFE suction line and lower to the midpoint of the well screen, if known, or slightly below the existing water level.
- Purge well until the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)

- Ensure that equilibrium is established by making three consecutive readings, where one casing volume is pumped between each reading.
- Collect VOA samples, if required, with a bailer following guidelines established within this appendix.
- Fill sample bottles for remaining parameters by allowing pump discharge to flow gently into the bottle with minimal entry turbulence. Pump discharge should be less than 500 mL/min. Cap each bottle as filled.
- Preserve the samples 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 complete the label with all necessary information.
- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.
- Complete chain-of-custody documents, field sheet, and field logbook (see Instruction F-1, "Documentation," in Appendix F).
- Allow system to drain, then disassemble. Decontaminate or replace tubing for next sampling.
- Close the well.

C.2.4.8 Air lift pump. Air lift pumps are usually used for developing or purging recovery wells rather than monitoring wells because of their ease of use and their ability to maintain moderate flow rates. Air lift pumps are not recommended for sampling monitoring wells. Because of the contact with well water and the source gas, the sample could be chemically altered. Depending on the source gas, the pH could be altered, oxidation and degassing of VOCs could occur, and lubricating oils from the air compressor could be introduced.

C.2.4.9 Low-flow (minimal-drawdown) sampling. This method uses any adjustable rate pump that can maintain low flow (typically less than 0.5 L/min) and minimize drawdown. Drawdown ideally should be less than <10 cm (4 in.) during the entire purging and sampling process. Typically used pumps include submersible centrifugal pumps, bladder pumps, and peristaltic pumps. Some pumps are better suited than others, given the well and aquifer constraints. Submersible pumps, for example, have reportedly had difficulty maintaining low flow in shallow 6- to 7.6-m (<20- to 25-ft) aquifers and may overheat under those circumstances. Additional equipment required includes an electronic water level indicator (accurate to 0.3 cm (± 0.01 ft), a multiparameter probe (dissolved oxygen, turbidity, redox potential, pH, temperature, and specific conductivity), and a flow-through cell compatible with the probe.

C.2.4.9.1 Applicability. Low-flow sampling is generally applicable in any instance where a representative nonturbid sample of the aquifer is desired. It is particularly appropriate in low-yield formations where the well would go dry if pumped at higher rates. It is also important to use low-flow techniques in wells that produce samples with high turbidity, which can result in high metals analytical results. Low-flow sampling induces a lower velocity through the screen, which entrains fewer particulates.

Another important advantage is that less purge water is produced, significantly decreasing the costs for handling and disposing of Investigation Derived Wastes (IDW). To implement low-flow sampling appropriately, the water level in the well being sampled needs to be monitored continuously, and the actual flow rate must be measured and adjusted to maintain drawdown in the well to less than 10 cm (4 in.).

C.2.4.9.2 Method summary and equipment. The pump must also be inserted into the well in a way that minimizes the disturbance of any sediments within the well casing, particularly those in the bottom of the well. In no circumstances should the water level probes or pumps be allowed to hit the bottom of the well before or during sampling. If possible the point where water enters the pump should be at least 0.6-0.9 m (2-3 ft) above the bottom of the well. The pump should be turned on at the lowest setting possible, and that setting should be raised gradually until a continuous flow is achieved from the discharge tube. During this time the water level needs to be monitored and the flow rate adjusted to maintain the drawdown to less than 10 cm (4 in.) if possible. In no circumstances should the water level be allowed to break suction in the pump. The pump is to be turned off when that happens and turned on again when sufficient recharge has occurred. If there is a stagnant column of water in the casing above the screen, it is essential that the water level not be allowed to come down to the screen. If this should appear imminent, the pump should be shut off and the well allowed to recover before purging continues. There are wells that even at pumping rates of 100-200 mL/min will not be able to recharge sufficiently. In those circumstances the sampling team needs to reassess the sampling goals for that location and determine whether there are other more appropriate ways to obtain a representative sample. To successfully monitor the representativeness of the water sample the most sensitive parameters (DO, redox potential, and turbidity) must be measured along with the traditional parameters of pH, temperature, and conductivity. To accurately measure DO and redox requires the use of a flow-through cell, which allows water to be pumped directly from the well and contact the probe(s) without contacting the atmosphere. There is no acceptable alternative to the flow-through cell for accurate measurement of DO and redox. Readings should be taken of all parameters every 3-5 minutes. If after three casing volumes have been removed from the well the turbidity is not <5, this should be noted in the log and the samples should be collected.

C.2.4.9.3 Sampling procedures. Recommended sampling procedures are as follows:

- Prepare the work area outside the well by placing plastic sheeting on the ground to avoid cross-contamination.

- Determine the saturated water column in the well using an electronic water level indicator. NOTE: Do not tag the bottom of the well since the total depth should be on the well construction diagram. Anything that hits the bottom of the well risks disturbing any fines that are there, and they may subsequently be entrained in the sample. Calculate the fluid volume in the casing and determine the amount of water to be removed for purging.

- Connect the pump tubing to the flow-through cell and connect the multiparameter probe to the cell.

- Purge well until the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium, and begin sampling. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)

- Establish equilibrium by making three consecutive readings, with 3 to 5 minutes between each reading.

- Detach the tubing from the flow-through cell and collect VOA samples first.
- Collect VOA samples, if required, following guidelines established within this appendix. Fill sample bottles for remaining parameters by allowing pump discharge to flow gently into the bottle with minimal entry turbulence. Pump discharge should be less than 500 mL/min. Cap each bottle as filled.
- Preserve the samples 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 complete the label with all necessary information.
- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.
- Complete chain-of-custody documents, field sheet, and field logbook (see Instruction F-1, "Documentation," in Appendix F).
- Allow system to drain, then disassemble. Decontaminate or replace tubing for next sampling.
- Close the well.

C.2.4.10 Emerging and innovative sampling procedures. All of the standard samplers described in this appendix for the sampling of ground water disrupt equilibrium during the sampling event to some degree. This may lead to obtaining a sample that is not representative of the actual environmental conditions or contaminant concentrations. New sampling devices and sampling procedures have been designed to minimize the disturbance to the medium during sampling, circumvent the need to purge the well, and minimize the influence on and any bias to the results that this disturbance, the stagnant water above or below the well screen, or fines contained within the monitoring well may impose. Recent studies have shown the application of diffusion samplers to the acquisition of samples for VOCs, a chemical parameter susceptible to loss from the disturbance imposed by purging and sampling (Vroblesky and Hyde 1997). The diffusion samplers are constructed of polyethylene bags containing deionized water that is strategically hung within the screened interval of the monitoring well and left for a predetermined time (e.g., 11-54 days). Polyethylene is shown to allow the transmission of various halogenated and aromatic VOCs, and is used as a semipermeable membrane to allow the diffusion of VOCs from the ground water to the deionized water contained within the bags. The sampler may also be designed to have multiple discrete cells that are vertically separated. This configuration allows the monitoring of VOC concentrations as they pertain to different depth intervals. After the sampling period, the diffusion samplers are collected from the wells by the attached strings, and the water is gently poured from the sampler into 40-mL VOA vials through the PTFE stopcock. Samples are then processed in the same manner as other sampling techniques. The diffusion samplers are commercially available; easy to deploy, retrieve, and sample; and an inexpensive alternative for the acquisition of VOC samples in ground water monitoring wells or other water bodies.

C.2.4.11 Decontamination procedures. All equipment that will enter the well must be decontaminated before entry. The inside surface of pumps and tubing apparatus must be decontaminated by drawing the decontamination solution through the equipment. Field measurement equipment such as water level indicators should be cleaned as described in Instruction E-6, Appendix E. If the sampling equipment is being

prepared for later use, it should be wrapped in cleaned foil. The sampling equipment should remain wrapped in this manner until immediately before use. Additional sampling devices may be needed onsite to ensure an adequate drying time. The requirement for dedicated equipment should apply to all bailers used for collecting samples. Bailers, other sampling equipment, and sample bottles must be physically separated from generators during transport and storage. Decontamination procedures for field equipment are discussed in Instruction E-6 (Appendix E).

C.2.4.12 Field control sample requirements. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They include blanks, replicates, and/or background (upgradient) samples. Quality Assurance (QA) samples are replicates sent to a referee (QA) laboratory and analyzed to evaluate the contractor's laboratory performance. Quality Control (QC) samples are blind replicates collected by the sampling team for analysis by the primary laboratory. A detailed discussion of field control samples is contained in Instruction G-2 (Appendix G).

C.2.4.13 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site setup and sample collection and handling as outlined previously and in Instruction F-1 of Appendix F.

C.3 Surface Water Sampling

C.3.1 Scope and application.

C.3.1.1 This instruction presents guidelines for collecting representative samples from surface water bodies. Surface water bodies can be classified into two primary types: flowing and standing. Flowing bodies include industrial effluent, municipal wastewater, rivers, sewers, leachate seeps, streams, or any other lotic water body. Standing bodies include lagoons, ponds, nonaqueous (e.g., surface impoundments), lakes, or any other lentic water body. Surface water samples can be collected from various depths of the water bodies using some of the techniques described herein. Instructions for sampling surface water bodies using the following techniques are included: hand-held bottle, dipper, pond sampler, peristaltic pump, Kemmerer sampler, weighted bottle, and Bacon bomb sampler.

C.3.1.2 Storm water runoff sampling is another type of surface water sampling done to acquire the qualitative and quantitative data necessary to complete National Pollution Discharge Elimination System (NPDES) storm water permits, and to determine the effectiveness of storm water management plans. NPDES permits are issued by the state for permanent areas that due to their purpose or mission (e.g., petroleum, oils, and lubricants (POL), runway, hazardous waste (HW) storage area) may pose a potential environmental risk from storm water runoff through the area. The permit should define the appropriate sampling locations and testing requirements based on the location and types of contaminant sources present and site topography. Sampling of storm water runoff is based on simple standard sampling techniques to acquire representative grab and composite samples from key runoff locations to monitor for visual properties and analytical chemistry parameters as defined by the NPDES permit. Further details on storm water runoff sampling requirements may be found in 40 CFR 122.21(g)(7), 40 CFR 136, and EPA/833/B-92/001.

C.3.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified in the HTRW technical planning process. The sampling strategy developed for a particular site will influence several project decisions, including but not limited to sampling locations, types of samples, sampling frequency, and sampling and analytical protocols. For instance, ecological risk assessment may require the need for colocated surface water and sediment samples. Sampling strategies may be significantly influenced by such factors as physical site constraints, safety, and cost, to name a few. The technical planning process that results in the development of the sampling strategy is critical because of the difficulty in acquiring representative surface water samples, the reduction of contaminant action levels, and the problems associated with trace level cross-contamination. Successful investigations of hazardous waste sites are highly dependent on an effective sampling scheme. Development of a sampling scheme to characterize a hazardous waste site should follow the fundamentals of the scientific approach. A successful sampling scheme requires a logical design to allow an evaluation of potential contaminants in relation to background conditions, vertical extent, horizontal extent, and mobility in various media. Regulations covering storm water runoff sampling require that it be performed during a qualifying storm event. The following criteria must be met for the storm event to qualify and the resulting data to be considered a valid data record: the storm event must be preceded by 72 hours of dry weather (rainfall <2.5 cm (<0.1 in.)), the variance in the duration and total rainfall of the storm event may not exceed 50 percent of the average storm event, the storm must produce greater than 2.5 cm (0.1 in.) of rain, and the storm must produce runoff.

C.3.2.1 Sampling locations. Sampling at hazardous waste sites is usually conducted in an attempt to identify contamination and to define its extent and variability. With such an objective, it is most logical to choose sample locations that will yield the most information about site conditions. However, other factors such as accessibility, sampling equipment requirements, and demands on the sampling team need to be considered when selecting locations. When a site is evaluated, sampling can be conducted by random, systematic, or biased sampling. Biased samples are those collected at locations that were chosen based on

historical information, knowledge about the behavior of the contaminants, and/or knowledge about the effects of the physical system on the fate of the contaminant. Random sampling depends on the theory of random chance probabilities to choose the most representative sample. Often biased and random sampling techniques can be used together to address an entire site thoroughly. Some samples may be biased to potentially contaminated areas or potentially impacted areas (e.g., downstream from discharge pipe). In areas less likely to be contaminated or areas with little available background information, random samples may be used to allow adequate assessment of the entire site. Due to the nature of the media, locations for surface water samples are restricted to locations within the water body under evaluation. However, variations of those within the water body may include depth, horizontal location, and time. Storm water runoff sampling locations are defined within the NPDES permit based on the mission and purpose of the evaluated areas and the site topography. Each sample location is defined by the facility name and outfall number.

C.3.2.2 Types of samples. The type of sample should be designated when selecting a sampling method. Surface water samples may be discrete (grab) or composite samples. A discrete (grab) sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected at one particular point in the sample matrix. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease. Composites are samples composed of two or more specific aliquots (discrete samples) collected at various sampling locations and/or different points in time. Analysis of this type of sample produces an average value and can, in certain instances, be used as an alternative to analyzing a number of individual grab samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix. Regulations covering storm water runoff sampling require that both grab and flow-weighted composite samples be collected. Grab storm water samples are discrete samples, normally collected within the first 30 minutes after the onset of runoff from the storm event or as soon as practicable, but no later than within the first 60 minutes of storm water runoff discharge. To comply with 40 CFR 136, grab sampling techniques are required for several potential analytical chemistry parameters (e.g., pH, temperature, VOCs, oil and grease, fecal coliform, fecal streptococcus, cyanide, total phenols, and residual chlorine). Flow-weighted composite samples are taken during the first 3 hours (or an entire event if less than 3 hours in length) of the storm event. It is a mixed sample from one location, combining discrete samples at specified time intervals in specific volumes that are proportional to the runoff flow. Details on the procedures for measuring or estimating rainfall and runoff flow rates should be referenced from EPA/833/B-92/001.

C.3.2.3 Suggested samplers. Each sampling technique presents various disadvantages and advantages for its application. For example, desired depth, tidal influences, sample disturbance, sample volume, chemical/physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique. The following sections discuss the advantages and disadvantages of each sampling technique.

C.3.2.4 Sample frequency. Determination of the number of samples needed to characterize a site depends upon sampling objectives and site-specific conditions. For example, if the objective of the event is to determine whether the site is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, the site is known to be contaminated and delineation of the contamination is the objective, a greater number of samples may be needed. In many cases, statistical considerations can be helpful in determining sampling strategy. It may also be necessary to strategically plan the timing of sampling. For example, industrial discharges may be more likely during working hours. Storm water runoff sampling frequency requirements are defined within the facility's NPDES permit.

C.3.3 Sample preservation and handling. Many of the chemical constituents and physicochemical parameters that are to be measured or evaluated in monitoring programs are not chemically stable; therefore, sample preservation is required. Appropriate preservation techniques for various parameters are specified in Appendix B. In addition, sample containers that should be used for each constituent or common set of parameters are specified in Appendix B. These preservation methods and sample containers are based on EPA/SW-846. Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2, "Packaging and Shipping Procedures," in Appendix F. Improper sample handling may alter the analytical results of the sample, causing the results to be invalid. Samples should be transferred in the field from the sampling equipment directly into the container that is required for that analysis or set of compatible parameters and in sufficient volumes, including number of sample containers, to allow appropriate analyses. The sample should then be preserved in the field as specified in Appendix B. Because of the low analytical detection limits that are required for assessment of ecological risk and other data uses, care must be taken when collecting the sample to avoid the loss of any contaminants. The samples for volatile analysis should be carefully transferred directly from the sample collection device to the sample container in order to minimize contaminant loss through agitation/ volatilization or adherence to another container. Samples should be collected in the order listed in Section C.3.3.1 When more than one container is required per parameter, the sample should be split equally among all containers until they are filled. Containers used to collect samples for organic analyses should not be prerinsed with water because of the possibility of preservation loss or the loss/gain of contaminants that may taint the analytical results.

C.3.3.1 Sample containers. When metals are the analytes of interest, HDPE containers with PTFE-lined polypropylene caps should be used. When organics are the analytes of interest, glass bottles with PTFE-lined caps should be used. Refer to Appendix B or the specific analytical method to designate an acceptable container. Containers should be cleaned based on the analyte of interest. Instruction E-6, "Decontamination Procedures," (Appendix E) contains additional information on appropriate glassware cleaning protocols. If precleaned bottles are used, the cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Refer to Appendix B for information on the required size, number, and type of sample containers. Samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. A preferred collection order for some common parameters follows:

- VOA
- POC
- POX
- TOX
- TOC
- Extractable organics
- Total metals
- Dissolved metals
- Phenols
- Cyanide
- Sulfate and chloride

- Turbidity
- Nitrate and ammonia
- Radionuclides

C.3.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and to reduce sorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and protection from light. Prepreserved sample containers are not recommended. Because of the potential loss of preservative if field errors occur and because different amounts of preservative may be necessary to bring the sample to the required pH, it is recommended to add the preservative to the container in the field and verify that the pH of the sample has been achieved. This information should be documented within field logbooks. The sampler should refer to Appendix B or the specific preservation method in EPA/SW-846 for the appropriate preservation technique.

C.3.3.3 Special handling for VOA samples. Water samples to be analyzed for purgeable organic compounds should be stored in 40-mL vials with septum inserts and screw caps. The septum should be placed on the sample vial so that the PTFE side is in contact with the sample. The 40-mL vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence that could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The septum is then applied and some overflow is lost, but air space in the bottle is eliminated. After the bottle is capped, it should be turned over and tapped to check for bubbles. If any bubbles are present, the procedure must be repeated. Care should be taken to ensure that no loss of preservative occurs, if applicable.

C.3.3.4 Special precautions for trace contaminant sampling. Contaminants can be detected in the parts per billion and/or parts per trillion ranges. Therefore, extreme care must be taken to prevent cross-contamination of these samples. The following general precautions should be taken when sampling:

- A clean pair of new disposable gloves should be worn each time a different location is sampled, and gloves should be donned immediately prior to sampling.
- To prevent cross-contamination between samples, it is suggested that the multiple vials from each sampling location be sealed in separate smaller plastic bags when the sampled medium is suspected of containing high concentrations of volatile organics.
- Sample containers filled with source or waste samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting and preserving, and activated carbon should be included in the bags to prevent cross-contamination.
- If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants should be lined with new, clean, plastic bags.

- If possible, one member of the field team should take all the notes and fill out sample tags, field sheets, etc., while the other members collect all of the samples.
- Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.
- Field personnel should use equipment constructed of PTFE, stainless steel, or glass that has been properly precleaned. PTFE or glass is preferred for collecting samples where trace metals are of concern.
- Adequate field control samples should be collected.

C.3.4 Sampling methods. Sampling instructions for the most common techniques for collecting surface water samples are presented in this section. Additional guidance on surface water sampling methods may be found in EM 200-1-2 and EPA/600/2-80/018. Prior to sample collection, water body characteristics (size, depth, flow) should be recorded in the field logbook. Sampling should proceed from downstream locations to upstream locations so that disturbance related to sampling does not affect the samples collected on the upstream side. In addition, if sediment samples are to be collected at the same locations as water samples, the water samples must be collected first. If the project requirements make it necessary to distinguish the concentration of metals in solution from the concentration of metals associated with solids, filtration of the surface water will be required. Filtration techniques are discussed in Instruction E-1 (Appendix E) of this manual. The factors that will contribute to the selection of a surface water sampler include the width, depth, and flow of the surface water body location being sampled, and whether the sample will be collected from the shore or a vessel. For flowing liquids an additional concern must be addressed. Tidal influence should be determined, and its influence on sample collection should be detailed in the sampling plan. At a minimum, the stage of the tide at the time of sample collection should be recorded. Consideration should be given to sampling at varied tidal stages. Samplers may encounter situations where rate of flow affects their ability to collect a sample. For fast-flowing rivers and streams, it may be nearly impossible to collect a midchannel sample at a specific point. Low- flowing streams and leachate seeps present the opposite problem. In these cases, the sampler should attempt to find a location where flow is obstructed and a pool is created. If this is not possible, the only way to obtain a sample may be to dig into the sediment with a decontaminated trowel to create a pooled area where the liquid will accumulate. However, this method is not recommended since the sample is likely to be highly turbid. If the banks are not sloping, sampling personnel may be able to collect the liquid directly into the sample bottle from the edge of the water body. In some instances where the liquid to be sampled cannot be reached, a pond sampler, by virtue of its extension capabilities, may be necessary. In these cases, the pond sampler should be assembled to ensure that sampling personnel are not in danger of falling into the water body being sampled. In cases where access is restricted, or data objectives require a sample taken from the middle of the water body, a boat, barge, or other stable working platform may be necessary. For a stream, channel, or river, the sample should be collected at middepth. For standing liquid, the sample should be collected just below the surface or at middepth. Specific sampling strategies may be altered depending on the contaminants of concern. For instance, when sampling for hydrocarbons or other light nonaqueous phase liquids, it may be better to sample at the surface. Once the sample is obtained, it should be transferred directly into the sample bottle. The sampling device should be decontaminated before the next sample is taken. If sampling below the water surface is required, some of the samplers discussed in the following sections will allow collection of discrete representative liquid samples at various depths. Proper use of the sampling device chosen includes slow lowering and retrieval of the sample, immediate transfer of the liquid into the sampling container, and notation in the logbook of the depth at which the sample was collected.

C.3.4.1 Hand-held bottle.

C.3.4.1.1 Applicability. Filling the sample containers directly is advantageous when the sample might be significantly altered during transfer from a collection vessel into another container. This would affect samples being collected for VOC analysis. The hand-held bottle is not applicable for samples required at depth.

C.3.4.1.2 Method summary and equipment. Samples from shallow depths can be readily collected by merely submerging the sample containers.

C.3.4.1.3 Sampling procedure. The recommended sampling procedure follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.
- Submerge the sample container with the cap in place with minimal surface disturbance so that the open end is pointing upstream.
- Allow the device to fill slowly and continuously using the cap to regulate the speed of water entering the bottle.
- Retrieve the sample container from the surface water with minimal disturbance.
- 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 sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.
- Record the information in the field logbook and complete the chain-of-custody form and field sheets (See Instruction F-1, "Documentation," in Appendix F).

C.3.4.2 Dippers and pond samplers. Method Reference: ASTM D 5358.

C.3.4.2.1 Applicability. Dippers and pond samplers prevent unnecessary contamination of the outer surface of the sample bottle that would otherwise result from direct immersion in the source. Dippers and pond samplers can be either reused or discarded. Discarding the samplers would eliminate the need for decontamination. With the pond sampler, samples can be obtained at distances as far as 3 m (10 ft) from the edge of the source, preventing the technician from having to contact the source physically. The tubular handle may bow when sampling very viscous liquids if sampling is not done slowly. Dippers and pond samplers perform similar functions, except that the length of the dipper is smaller.

C.3.4.2.2 Method summary and equipment. The pond sampler consists of an adjustable clamp attached to the end of a two- or three-piece telescoping aluminum or fiberglass pole that serves as the handle. The clamp is used to secure a sampling beaker (Figure C-4).

C.3.4.2.3 Sampling procedure. The recommended sampling procedure follows:

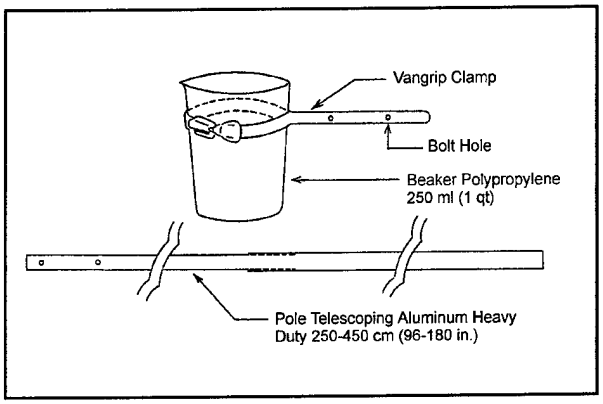


Figure C-4. Pond sampler

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.
- Assemble the dipper or pond sampler. If appropriate, make sure that the sample container and the bolts and nuts that secure the clamp to the pole are tightened properly.
- Collect samples by slowly submerging the precleaned dipper or pond sampler with minimal surface disturbance. Make sure that the open end is pointed upstream.
- Retrieve the dipper or pond sampler from the surface water with minimal disturbance.
- Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the edge of the dipper/sampler.
- Empty the sampler slowly, allowing the sample stream to flow gently down the side of the bottle with minimal entry turbulence.
- Continue delivery of the sample until the bottle is filled.
- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with an appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.
- Record the information in the field logbook and complete the chain-of-custody documents and field sheets (See Instruction F-1, "Documentation," in Appendix F).
- Properly clean and decontaminate the equipment prior to reuse or storage.

C.3.4.3 Peristaltic pump. Method Reference: EPA/600/4-84/076

C.3.4.3.1 Applicability. An advantage of the peristaltic pump is its design, which isolates the sample from the moving part of the pump and allows for easy decontamination by removal or replacement of the

flexible tubing. This method can both extend the lateral reach of the sampler and allow sampling from depths below the water surface. Disadvantages of these pumps include their low pumping rates and their limited height of intake lift (less than 6 m (20 ft)). These pumps also require an outside power source and, like other suction pumps, are not suitable for collecting samples for VOC analysis because of potential degassing effects.

C.3.4.3.2 Method summary and equipment. Peristaltic pumps displace fluid by mechanical peristalsis. A flexible sampling tube is mounted around the pump chamber, and rotating rollers compress the tubing, forcing fluid movement ahead (the peristaltic effect) and inducing suction behind each roller (Figure C-3).

C.3.4.3.3 Sampling procedure. The recommended sampling procedure follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.

- Install clean, medical-grade silicone tubing in the pump head, as instructed by the manufacturer. Attach the silicone tubing to the glass tubing outlet from the sample bottle. If the sampling device is not constructed as shown in Figure C-3 and the sample bottle is filled directly from the discharge line of the peristaltic pump, the sample will be in direct contact with the intake tubing, the pump head, and the discharge tubing prior to release to the sample container. In this situation, PTFE tubing must be used for the discharge line to avoid cross-contamination of the samples from contaminant leaching that would occur from other "less inert" tubing.

- Select the length of suction intake tubing necessary to reach the required sample depth and attach it to the intake side of the sample bottle. Heavy-wall PTFE or a diameter equal to the required pump tubing will suit most applications. (A heavier wall will allow for a slightly greater lateral reach.)

- If possible, allow several liters of sample to pass through the system before actual sample collection. Collect this purge volume and return it to the source after the sample aliquot has been withdrawn.

- Collect VOA samples, if required, with an alternative sampling device following guidelines established in this appendix.

- Fill the necessary sample bottles by allowing pump discharge to flow gently down the side of bottle with minimal entry turbulence. Cap each bottle as filled.

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

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

- Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.

- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.

- Record the information in the field logbook and complete the chain- of-custody documents and field sheets (See Instruction F-1, "Documentation," in Appendix F).

- Allow system to drain, then disassemble it. Decontaminate tubing if necessary; otherwise discard appropriately.

C.3.4.4 Kemmerer sampler. Method References: ASTM D 4136 and EPA/540/P-91/005, Standard Operating Procedure (SOP) #2013, "Surface Water Sampling."

C.3.4.4.1 Applicability. The Kemmerer sampler is a practical method for collecting discrete, at-depth samples where the collection depth exceeds the lift capacity of pumps. The use of the Kemmerer sampler is limited, however, because it is typically constructed of brass.

C.3.4.4.2 Method summary and equipment. The Kemmerer sampler is a messenger-activated water sampling device that is used to sample water from a specific depth (Figure C-5). In the open position, water flows easily through the device. Once the device is lowered to the desired depth, a messenger is dropped down the sample line tripping the release mechanism and closing the container. In the closed position, the bottle is sealed at the top and bottom, isolating the sample during retrieval.

C.3.4.4.3 Sampling procedure. The recommended sampling procedure follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.

- Inspect Kemmerer sampler to ensure that sample drain valve is closed (if equipped).

- Measure and mark sampler line at desired sampling depth.

- Open bottle by lifting top stopper-trip head assembly.

- Gradually lower bottle until desired sample depth is reached.

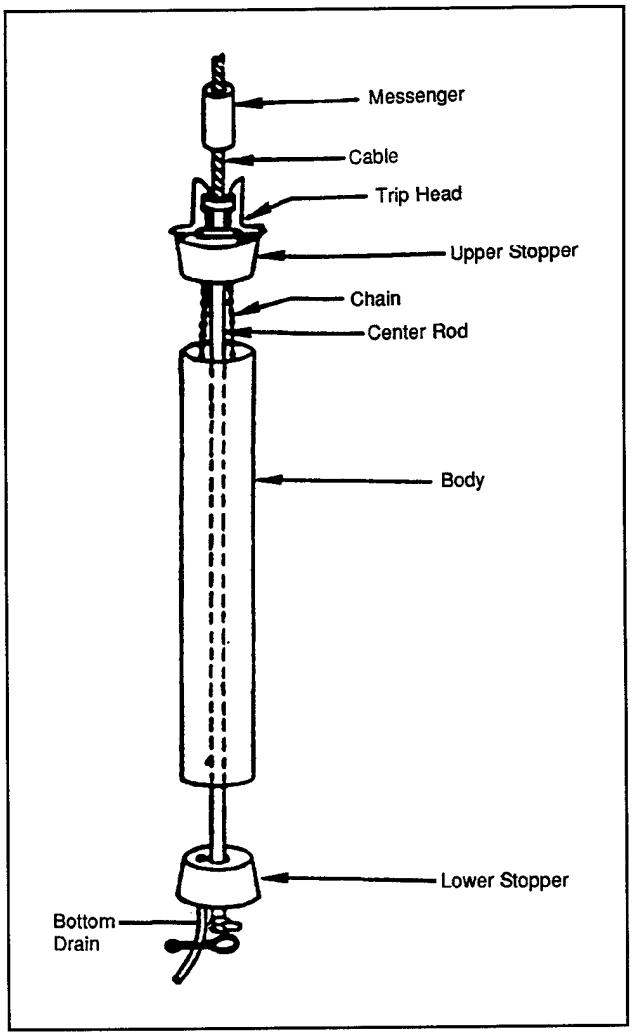


Figure C-5. Kemmerer sampler

- Place messenger on sample line and release.
- Retrieve sampler; hold sampler by center stem to prevent accidental opening of bottom stopper.
- Rinse or wipe off exterior of sampler body.
- Recover sample by grasping lower stopper and sampler body with one hand (gloved), and transfer sample by either lifting top stopper with other hand and carefully pouring contents into sample bottles or holding drain valve (if present) over sample bottle and opening valve.
- Allow sample to flow slowly down the side of the sample bottle with minimal disturbance.
- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.
- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.
- Record the information in the field logbook and complete all chain-of-custody records and field sheets (See Instruction F-1, "Documentation," in Appendix F).
- Decontaminate sampler.

C.3.4.5 Weighted bottle. Method References: ASTM D 4057 and ASTM E 300.

C.3.4.5.1 Applicability. The weighted bottle can be used to obtain samples from a specific depth. The glass construction of the sampler can make the use of this sampler more desirable than the Kemmerer in some sampling situations.

C.3.4.5.2 Method summary and equipment. The weighted bottle can be used for collecting representative samples from a specific depth. The sampler consists of a glass bottle, a weighted sinker, a bottle stopper, and a line that is used to lower and raise the sampler during sampling. Once the sampler is lowered to the desired sampling depth, the stopper is opened, and the bottle is filled and retrieved to the surface.

C.3.4.5.3 Sampling procedure. The recommended sampling procedure follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross- contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.

- Assemble the weighted bottle sampler as shown in Figure C-6.
- Measure and mark the sampler line at the desired sampling depth.
- Lower the sampling device to the predetermined depth.
- When the sampler is at the required depth, pull out the bottle stopper with a sharp jerk of the sampler line and allow the bottle to fill completely. (This is usually evidenced by the cessation of air bubbles.)
- Retrieve the sampler.
- Rinse or wipe off the exterior of the sampler body.

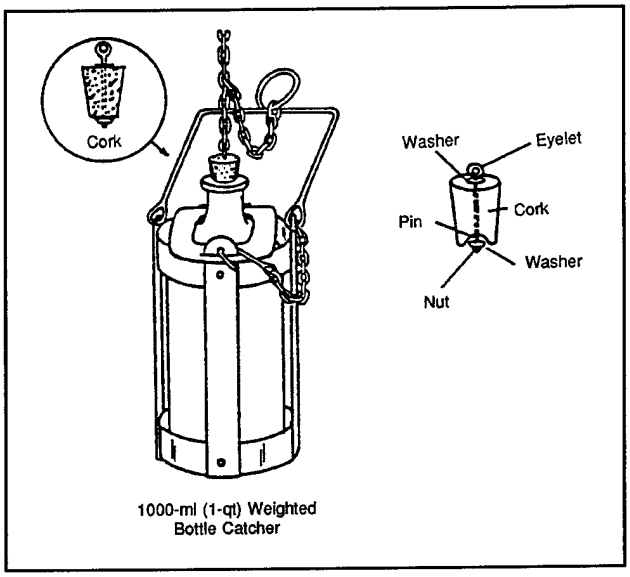


Figure C-6. Weighted bottle

- Allow sample to flow slowly down the side of sample bottle with minimal disturbance.
- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.
- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.
- Record the information in the field logbook and complete all chain-of-custody records and field sheets (see Instruction F-1, "Documentation," in Appendix F).
- Decontaminate sampler.

C.3.4.6 Bacon bomb sampler. Method Reference: EPA/540/P-91/005, SOP #2013.

C.3.4.6.1 Applicability. The Bacon bomb sampler is a widely used, commercially available sampler, designed for sampling petroleum products and viscous liquids (Figure C-7). It is very useful for sampling larger storage tanks because the internal collection chamber is not exposed to a product until the sampler is triggered. It is useful in collecting samples at various vertical locations. Like the weighted bottle sampler, the Bacon sampler remains unopened until it reaches the desired sampling depth. The Bacon sampler is difficult to decontaminate, and it is difficult to transfer the sample into the sample bottles. The possibility of aerating the sample exists if the sampler does not completely fill with water and air is entrapped in the sampler during retrieval.

C.3.4.6.2 Method summary and equipment. The Bacon bomb sampler is constructed of brass or stainless steel and is available in two sizes: 37 mm (1.5 in.) or 87 mm (3.5 in.) in diameter. Samplers range in volume from 0.1 L to 1 L (4 oz to 32 oz). The Bacon bomb sampler is equipped with a trigger that is spring loaded. When opened, the trigger allows liquid to enter the collection chamber. When the trigger is released, liquid is prevented from flowing into or out of the collection chamber.

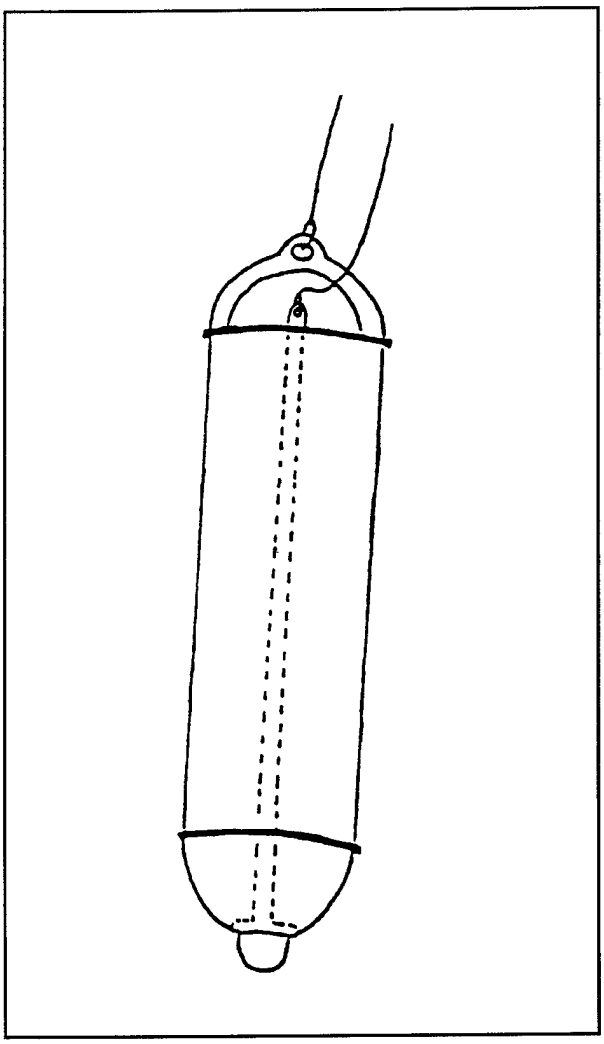


Figure C-7. Bacon bomb sampler

C.3.4.6.3 Sampling procedure. The recommended sampling procedure follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.
- Measure and mark the sampler line at the desired sampling depth.
- Lower the Bacon bomb sampler carefully to the desired sampling depth, allowing the line for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line taut.
- Release the trigger line and retrieve the sampler.
- Transfer the sample to the sample bottles by pulling on the trigger. Allow the sample to flow down the side of the sample bottle with minimal disturbance.
- Preserve the sample as necessary and verify that the pH is sufficient for the criteria.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.
- Place filled sample containers on ice immediately, along with the required trip blank, if analyzing for VOCs.

- Record the information in the field logbook and complete all chain-of-custody records and field sheets (see Instruction F-1, "Documentation," in Appendix F).

- Decontaminate the sampler.

C.3.4.7 Storm water runoff sampling techniques.

C.3.4.7.1 Applicability. Grab and flow-weighted composite samples are required to complete NPDES storm-permit requirements.

C.3.4.7.2 Method summary and equipment. Samples of storm water runoff are taken into the sample containers directly, or a bucket and transferred to the appropriate sample bottle containers.

C.3.4.7.3 Grab sampling procedure. The sampling procedure is as follows:

- After rain begins to fall, record the date and time rain started to produce storm water runoff, facility number, outfall number, height of water in a measurement device (i.e., rain gauge), sample number, sample type, other items as directed on the NPDES storm water field sheet (see EPA/833/B-92/001).

- Lower the sample container or stainless steel or PTFE sampling bucket into the center of the runoff flow where turbulence is at a maximum. Hold the sample container so the opening faces upstream. Avoid stirring up bottom sediments and keep sample free of uncharacteristic floating debris.

- Allow the device to fill, using care not to overfill the bottle.

- 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.

- Conduct visual observations as appropriate and record them in field log book.

- Label the sample bottle with an appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately along with the required trip blank, if analyzing for VOCs.

- Record the information in the field logbook and complete the chain-of-custody form and field sheets (See Instruction F-1, "Documentation," in Appendix F).

C.3.4.7.4 Flow-weighted composite sampling procedure. Flow-weighted composite sampling techniques are appropriate for most chemical parameters, with the exception of those noted as requiring grab sampling techniques within C.3.2.2 and 40 CFR 136. The aliquots for flow-weighted composite samples must be collected within the first 3 hours (or during the entire event if the storm is less than 3 hours). Equal aliquots may be collected at the time of sampling and then flow-proportioned and composited in the laboratory, or the aliquots taken may be based on the flow rate at the time of sample collection and composited in the field. Regulations require that a minimum of 15 minutes must separate the collection of

each sample aliquot, and that a minimum frequency of three sample aliquots within each hour of discharge be maintained. To help meet regulatory requirements, suggest sample aliquots be collected at 20-minute intervals. Use the following procedure:

- After rain begins to fall, record the date and time rain started to produce storm water runoff, facility number, outfall number, height of water in the measurement device (i.e., rain gauge), sample number, sample type, other items as directed on the NPDES storm water field sheet (see EPA/833/B-92/001).

- Determine and record runoff flow at this time as noted within EPA/833/B-92/001. Determine appropriate volume for sample aliquot.

- Lower the sample container (for lab compositing) or stainless steel or PTFE sample aliquot measuring device (for onsite compositing) into the center of the runoff flow where turbulence is at a maximum. Record the time and water depth. Hold the sample container so the opening faces upstream. Avoid stirring up bottom sediments and keep sample free of uncharacteristic floating debris.
- Allow the device to fill with appropriate volumes based on flow observed, or suggest a minimum of 1000 mL be acquired for each aliquot (for lab compositing). NOTE: The laboratory should also be consulted to determine the overall sample volumes necessary for the required analyses to ensure that sufficient volumes of individual sample aliquots are collected to support the composite sample.

- Repeat the steps for bullets 2-4 for each aliquot of the composite sample, retaining each storm water sample aliquot in separate, labeled sample containers. As stated in Section C.3.4.7.4, regulations dictate a minimum frequency of three sample aliquots within each hour of the storm event for the first 3 hours or duration of the storm event, if less than 3 hours.

- If field compositing is performed and after the sample aliquots have been collected, combine appropriate volumes of sample aliquots into stainless steel or PTFE bucket to create the flow-weighted composite sample. Fill appropriate sample bottles with the composite sample mixture, using care not to overfill the bottle.

- Preserve the sample if 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.

- Conduct visual observations as appropriate and record them in field logbook.

- Label the sample bottle with an appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately.

- Record the information in the field logbook and complete the chain-of-custody form and field sheets (see Instruction F-1, "Documentation," in Appendix F).

C.3.4.8 Emerging and innovative sampling procedures.

C.3.4.8.1 Disadvantages of standard samplers. All of the standard samplers presented previously for the sampling of surface water disrupt equilibrium during the sampling event. This may lead to obtaining a sample that is not representative of the actual environmental conditions or contaminant concentrations. These routine sampling techniques also acquire a one-time sample, which reflects only environmental conditions at the time of sampling, and they are unable to assess episodic contamination. Additionally, there is little understanding of the correlation between routine surface water sample results and the concentrations of truly dissolved or bioavailable contaminants. Many of the Applicable, Relevant, and Appropriate Requirements (ARARs) for surface water, which are aquatic toxicity data or water quality criteria, are based on dissolved contamination concentrations. Therefore, the use of standard sampling techniques to generate data for comparison with these criteria may not be appropriate.

C.3.4.8.2 New sampling devices and procedures. New sampling devices and sampling procedures have been designed to minimize the disturbance to the medium during sampling to minimize any bias in the results. Recent studies have shown the application of semipermeable membrane devices (SPMDs) to the sampling of surface water bodies (Petty et al. 1995; Ellis et al. 1995). This type of sampler is designed to mimic the bioconcentration process by capturing only the dissolved/bioavailable contaminant concentrations and is an alternative to performing tissue analyses of various species or aquatic organisms. It is also unique in that it assesses episodic contamination, due to the residence time within the medium under investigation. Although the use of this technique may not be fully accepted by a data user or regulating authority without some initial redundancy to routine procedures, the advantages it offers are compelling. SPMDs are commercially available; easy to deploy, retrieve, and sample; and very low maintenance. They are also able to detect contaminants at lower concentrations than routine surface water samples due to their ability to concentrate the contaminants within the lipid phase, as well as extending residence times, which may further enhance contaminant detection at low levels.

C.3.4.8.3 SPMDs. SPMDs (Figures C-8 and C-9) are constructed of thin-walled nonporous polymer lay-flat tubes (of low-density polyethylene, polypropylene, PVC, or silicone) containing a large molecular weight (• 600 daltons) nonpolar liquid (of neutral lipids - such as triolein, or silicone fluids) as a sequestered medium. The SPMD is housed within a protective shroud to avoid damage to the SPMD during sampling events. The semipermeable membrane (i.e., high-density polyethylene) film has pore sizes of approximately 10 • , which effectively control the rate of dissolved contaminants uptake into the device. The SPMD capacity to sequester organic contaminants is dependent on the lipid/water partition coefficient K_{LW} for that organic target analyte. This K_{LW} value can be approximated from the value for the octanol/water partition coefficient K_{ow} for the target analyte. SPMDs may be suspended to a specified depth (e.g., 0.5 or 1 m) of the surface water body, secured by floats and anchors for a period of several days to weeks. In general, the rate of contaminant uptake increases as water temperature increases and decreases as the device is biofouled by aquatic organisms. If water is clear and photosensitive compounds are being evaluated, the SPMD should be shaded to reduce contaminant loss through degradation. Heterotrophic growth may be minimized by periodic treatment (dipping) of the SPMD into a biocide, and/or the protective shroud may be manufactured of materials (i.e., copper screen) that inhibit biofouling. Post sampling, the SPMDs are collected and placed in clean jars or cans on ice for cooling. Ship SPMD samples to the laboratory within 24 hours. Holding time studies for SPMDs have been sporadic and have not assessed all potential contaminants. However, one study determined that freezing of SPMDs caused no loss of herbicide concentrations during 6 months.

C.3.4.8.4 SPMD uses. SPMDs may be used to screen for polynuclear aromatic hydrocarbons (PAHs), organochlorine pesticides, polychlorinated biphenyls (PCBs), herbicides, methyl mercury complexes, alkylated selenides, etc. Unfortunately, the analytical chemistry and data reduction procedures used for SPMDs are more labor intensive than routine environmental samples. Generally, the analytical procedures involve the following: initial cleaning and integrity inspection of the membrane; spiking SPMD media with

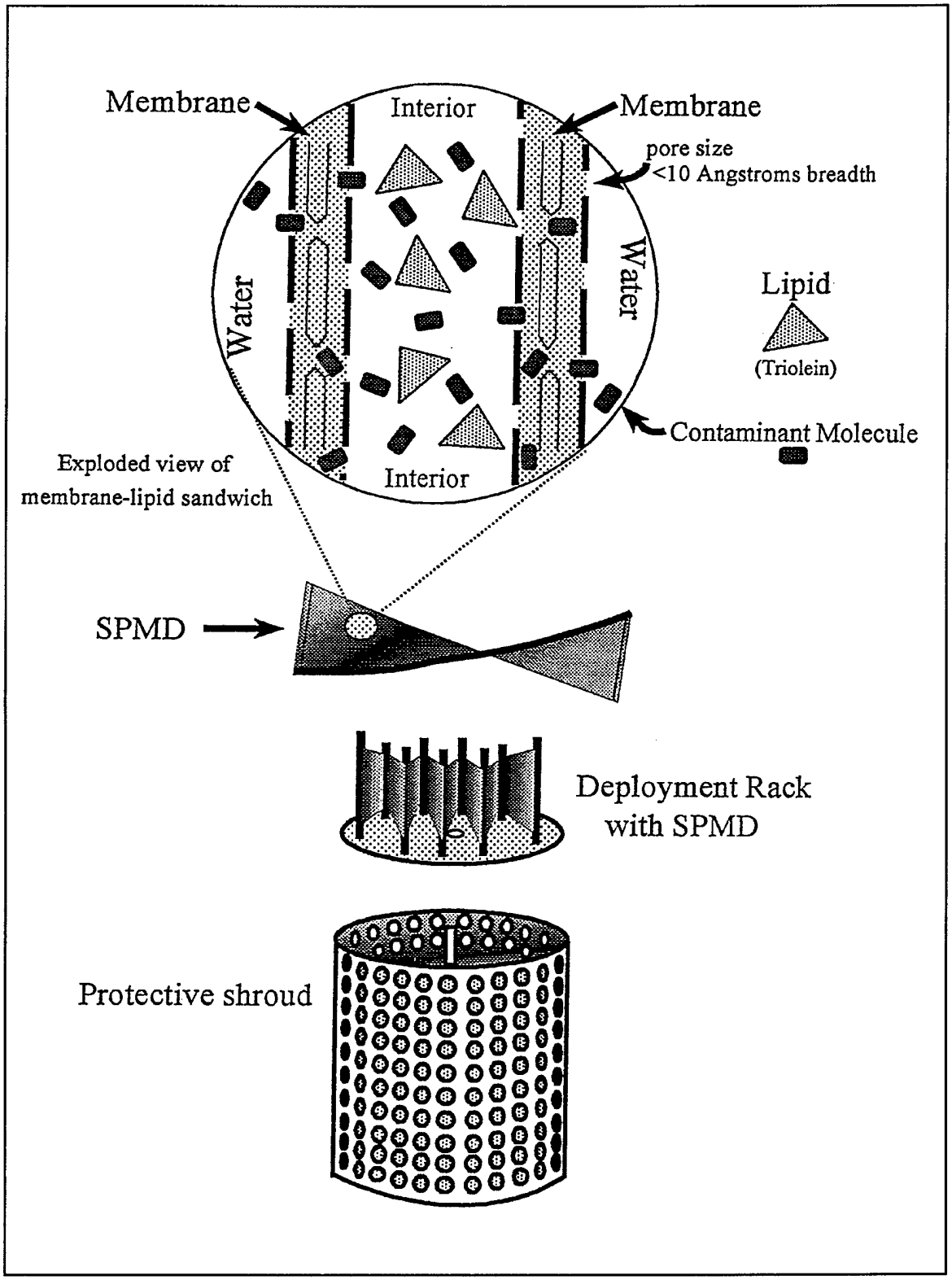


Figure C-8. Schematic of a semipermeable membrane device (SPMD)

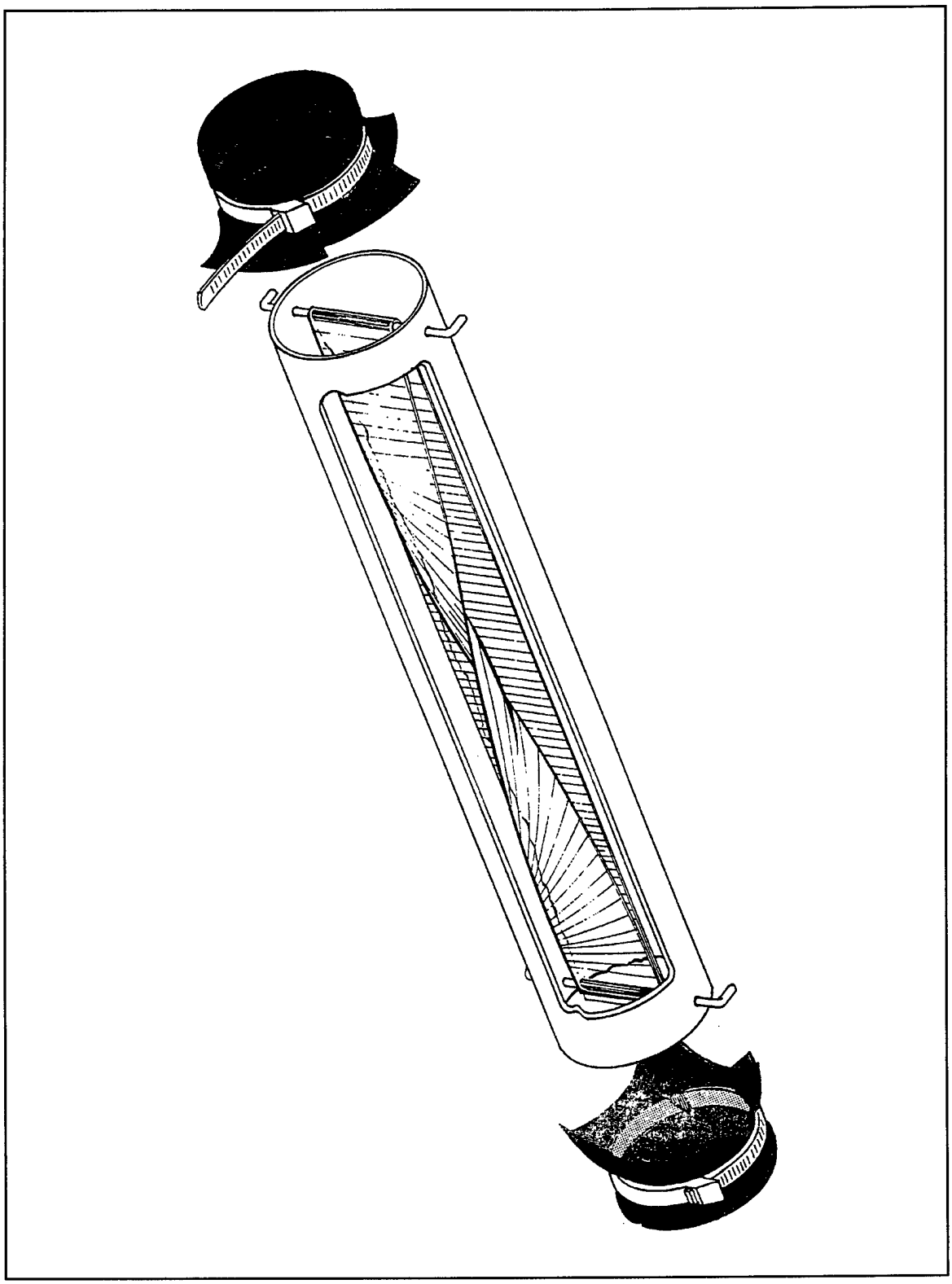


Figure C-9. Cutaway representation of an SPMD and protective shroud

surrogates or internal standards; placing SPMD in an appropriate solvent (i.e., hexane) to perform dialysis; undergoing sulfur cleanup of dialysates (if applicable); performing size exclusion chromatography (i.e., gel permeation column); performing additional chromatographic cleanups (florisil, silica gel); and performing final solvent exchange (if applicable) and solvent reduction procedures. Finally the extract undergoes analysis by gas chromatograph or high-performance liquid chromatograph configured with appropriate detectors. Currently, a major disadvantage of SPMDs is that few environmental laboratories have any experience with dialysis procedures. Concentrations of contaminants from SPMD data are calculated with information from these partitioning coefficients, the uptake rate constant, and exposure times. Refer to Petty et al. (1995) and Ellis et al. (1995) for details on the mathematical model used for this calculation.

C.3.5 Decontamination procedures. All equipment that will enter the water must be decontaminated prior to its entry. The inside surface of pumps and tubing apparatus must be decontaminated by drawing the decontamination solution through the equipment. Sampling equipment should be decontaminated, as described in Instruction E-6 (Appendix E). The sampling equipment should be placed in plastic bags until immediately prior to use. Additional sampling devices may be needed onsite to ensure an adequate drying time. During transport and storage, sampling equipment and sample bottles must be physically separated from engines/ generators that are used to power some sampling equipment.

C.3.6 Field control samples requirements. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They include blanks, replicates, and/or background (upgradient) samples. QA samples are replicates sent to a referee (QA) laboratory and analyzed to evaluate the contractor's laboratory performance. QC samples are blind replicates collected by the sampling team for analysis by the primary laboratory. A detailed discussion of field control samples is presented in Instruction G-2 (Appendix G).

C.3.7 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site setup, sample collection, and handling as outlined above and in Instruction F-1 of Appendix F.

C.4 Potable Water Sampling

C.4.1 Scope and application. This instruction presents guidelines for collecting representative potable water (tap water) samples. Discussions are based on the assumption that a supply tap is available for sampling the selected location, for example, a residence. Under this assumption the only applicable sampling method would be the hand-held bottle. The sampling methods discussed in Instruction C-2, "Ground Water Sampling," or C-3, "Surface Water Sampling," should be reviewed if other sampling methods are required for collecting a sample. Discussions presented in this section are a review of the protocols and procedures that should be used when collecting water samples from a tap.

C.4.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified in the HTRW technical planning process. The sampling strategy developed at a particular site will influence several project decisions, including, but not limited to, sampling locations, type of samples, sampling frequency, and sampling and analytical protocols. Sampling strategies may be significantly influenced by such factors as physical site constraints, safety, and cost, to name a few. The technical planning process that results in the development of the sampling strategy is critical because of the difficulty in acquiring representative samples, the reduction of contaminant action levels, and the problems associated with trace level cross-contamination. Successful investigations of hazardous waste sites are highly dependent on an effective sampling scheme. Development of a sampling scheme for purposes of characterizing a hazardous waste site should follow the fundamentals of the scientific approach. A successful sampling scheme requires a logical design to allow an evaluation of potential contaminants in relation to background conditions and contaminant degradation in various media.

C.4.2.1 Sampling locations. Potable water is usually sampled in an attempt to discover contamination and to define its variability. With such an objective, it is most logical to choose sample locations that will yield the most information about the water supply system. When a site is evaluated, sampling can be conducted by random, systematic, or biased sampling. Biased samples are those collected at locations that were chosen based on historical information, knowledge about the behavior of the contaminant(s), and/or knowledge about the effects of the physical system on the fate of the contaminant. Random sampling depends on the theory of random chance probabilities to choose the most representative sample. Potable water samples may also be collected for evaluating contamination in a particular well, assessing contamination due to a water piping system, or identifying the need for alternate water supply systems. When residential wells are sampled, the sample tap should not be located after a household purification system (i.e., water softening or filtration). In these cases an outdoor tap may have to be sampled. During assessment of water piping systems, contamination of both the hot and cold water sources should be considered. Depending upon the project data quality objectives (DQOs) and/or tap configuration, this may entail the acquisition of individual hot and cold water source samples, or opening both taps simultaneously during the sampling activity. Often biased sampling techniques are used to identify potentially contaminated or impacted areas. Water taps are stationary and are typically sampled for purposes of evaluating drinking water regulations or contaminant impact on local drinking water supplies. Selection of a sampling location is an investigation objective.

C.4.2.2 Type of sample. The type of sample should be designated when selecting a sampling method. Potable water samples are typically discrete samples. A discrete (grab) sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected at once and at one particular point in the sample matrix. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

C.4.2.3 Suggested samplers. The sample container is normally used to collect a potable water sample. Use of additional sampling equipment is not recommended. Sample disturbance, sample volume, and chemical/physical reactivity between potential contaminants and the sampling container should be considered when collecting the potable water sample.

C.4.2.4 Sample frequency. Determination of the number of samples needed to characterize a site is also dependent upon the objectives and site-specific conditions. For example, if the objective of the event is to determine whether the site is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, additional factors influencing contamination are the objectives, a greater number of samples may be needed. Timing for collecting samples may also be crucial. In many cases statistical considerations can be helpful in determining sampling strategy.

C.4.3 Sample preservation and handling. Because many of the chemical constituents and physicochemical parameters that are to be measured or evaluated in potable water monitoring programs are not chemically stable, sample preservation is required. Appropriate preservation techniques for various parameters and sample containers that the sampler should use for each constituent or common set of parameters are specified in Appendix B. These preservation methods and sample containers are based on EPA/SW-846. Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2, "Packaging and Shipping Procedures," in Appendix F. Improper sample handling may alter the analytical results of the sample, causing the results to be invalid. Samples should be collected in the container that is required for that analysis or set of compatible parameters and be in sufficient volumes to allow the appropriate analyses. The sample should then be preserved in the field as specified in Appendix B. Because of the low analytical detection limits that are required for assessment of drinking water standards and risk assessment data uses, care must be taken when collecting the sample to avoid the loss of any contaminants. Samples for volatile analysis should be taken in a manner that minimizes contaminant loss through agitation/volatilization. Samples should be collected in the order of the parameters listed in Section C.4.3.1. When more than one container is required per parameter, the sample should be equally split among all containers until they are filled. Containers used to collect samples for organic analyses should not be prerinsed with water because of the possibility of preservation loss or the loss/gain of contaminants that could taint the analytical results.

C.4.3.1 Sample containers. When metals are the analytes of interest, high-density polyethylene containers with PTFE-lined polypropylene caps should be used. When organics are the analytes of interest, glass bottles with PTFE-lined caps should be used. Refer to Appendix B or the specific analytical method to designate an acceptable container. Containers should be cleaned based on the analyte of interest. Instruction E-6, "Decontamination Procedures," Appendix E, contains additional information on appropriate glassware cleaning protocols. If precleaned bottles are used, the cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Refer to Appendix B for information on the required size, number, and type of sample containers. Samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. A preferred collection order for some common parameters follows:

- VOA
- POC
- POX
- TOX

- TOC
- Extractable organics
- Total metals
- Dissolved metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionuclides

C.4.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and reduce sorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and protection from light. Prepreserved sample containers are not recommended. Because of the potential for preservative loss if field errors occur, or if different amounts of preservative may be necessary to bring the sample to the required pH, it is recommended to add the preservative to the container in the field and verify that the pH of the sample has been achieved. This information should be documented within field logbooks. The sampler should refer to Appendix B or the specific preservation method in EPA/SW-846 for the appropriate preservation technique.

C.4.3.3 Special handling for VOA samples. Water samples to be analyzed for purgeable organic compounds should be stored in 40-mL septum vials with screw caps. Septa should be placed on the sample bottle so that the PTFE side is in contact with the sample. The 40-mL vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence that could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The septum is then applied and some overflow is lost, but air space in the bottle is eliminated. The bottle is then capped and should be turned over and tapped to check for bubbles. If any bubbles are present, the procedure must be repeated. Care should be taken to ensure that no loss of preservative occurs, if applicable.

C.4.3.4 Special precautions for trace contaminant sampling. Contaminants can be detected in the parts per billion and/or parts per trillion ranges. Therefore, extreme care must be taken to prevent cross-contamination of these samples. The following general precautions should be taken when sampling:

- A clean pair of new, disposable gloves should be worn each time a different location is sampled and gloves should be donned immediately prior to sampling.

- Sample containers for source samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting, preserving, tagging, etc.

- If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples shall be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag with activated carbon to minimize cross-contamination potential.

- If possible, one member of the field team should take all the notes, fill out sample tags, field sheets, etc., while the other members collect all of the samples.

- Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.

- Field personnel should use equipment constructed of PTFE, stainless steel, or glass that has been properly precleaned. PTFE or glass is preferred for collecting samples where trace metals are of concern.

- Adequate field control samples should be collected.

C.4.4 Sampling methods. When potable water is being sampled, utmost care must be taken to ensure that samples are representative of the water being sampled. This is important not only from a technical and public health perspective, but also from a public relations standpoint. Poor sampling techniques may result in incorrect results (either by not detecting a compound that is present or by contaminating the sample and falsely indicating a compound that is not present). If incorrect results are disclosed to the public, it may be impossible to change public opinion when correct results are reported. As discussed in Appendix C-2, "Ground Water Sampling," potable water wells must be purged before the sample is collected. This procedure ensures that water representative of the formation or source is sampled. The tap should be opened and allowed to flow to purge the system. Sampling should be performed after the DO, pH, conductivity, temperature, turbidity, and redox potential (if necessary) have reached equilibrium. The initial purging procedure ensures that any contaminants that might have entered the area of the tap from external sources have been avoided. Potable water samples should be representative of the water quality within the household or office under investigation. The sampling tap must be protected from exterior contamination associated with being too close to the sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collecting procedure since it is difficult to place a bottle under a low tap without grazing the neck interior against the outside faucet surface. Leaking taps that allow water to flow from around the stem of the valve handle and down the outside of the faucet or taps in which water tends to run up on the outside of the lip are to be avoided as sampling locations. Aerator, strainer, and hose attachments on the tap must be removed before sampling. These devices can harbor a bacterial population if they are not cleaned routinely or replaced when worn or cracked. Whenever a steady stream of water cannot be obtained from taps, after such devices are removed, a more suitable tap should be sought. Taps where the water flow is not steady should be avoided because temporary fluctuation in line pressure may cause sheets of microbial growth or scale that are lodged in some pipe section or faucet connection to break loose. A smooth-flowing water stream at moderate pressure without splashing should be obtained. Then, without changing the water flow, which could dislodge some particles in the faucet, the samples can be collected. Occasionally, samples are collected to determine the contribution of transmission pipes, water

coolers, water heaters, etc., to the quality of water in private residences, offices, etc. The purpose of these investigations may be to determine if metals, e.g., lead, are being dissolved into the water supply. In these cases, it may be necessary to ensure that the water source has not been used for a specific time interval, e.g., over a weekend or a 3- or 4-day holiday period. Samples collected may consist of one sample of the initial flush and another sample after the indicator parameters have reached equilibrium. Regardless of the type of sample bottle being used, the bottle septum and/or cap should not be placed on the ground or in a pocket. Instead, the bottle should be held in one hand and the septum and/or cap in the other, using care not to touch the PFTE side of the septa or inside of the cap. Exercise care not to lose the PTFE liner in certain bottle caps. Contaminating the sample bottle with fingers or permitting the faucet to touch the inside of the bottle should be avoided. Sample bottles should not be rinsed before use. When filling any container, care should be taken not to splash drops of water from the ground or sink into either the bottle or cap. To avoid dislodging particles in the pipe or valve, the stream flow should not be adjusted while sampling. Name(s) of the resident or water supply owner/operator and the resident's exact mailing address, as well as his or her home and work telephone numbers, should always be obtained. This information is required to inform the residents or water supply owner/operators of the results of the sampling program.

C.4.4.1 Hand-held Bottle.

C.4.4.1.1 Applicability. Filling the sample containers directly is advantageous when the sample might be significantly altered during transfer from a collection vessel into another container. This would affect samples collected for VOC analysis.

C.4.4.1.2 Method summary and equipment. Samples can be readily collected by directly filling the sample containers.

C.4.4.1.3 Sampling procedure. The sampling procedures previously discussed in this section should be addressed, if appropriate. The following are additional sampling procedures:

- If applicable, place plastic sheeting on the ground surface to prevent cross- contamination of samples.
- If applicable, remove aerator, strainer, and hose attachments on the tap before sampling.
- Purge well or source and tap lines initially at a high flow rate for a minimum of 5 to 10 minutes. Then adjust the flow to low to moderate and verify that the DO, pH, specific conductance, temperature, and turbidity are each at equilibrium. Equilibrium is established as follows: ±10 percent for DO, ±0.2 pH units, ±3 percent for specific conductance, ±1 degree Celsius for temperature, and ±10 percent for turbidity. (If redox potential is used as an indicator, ±10 mV should be used as the stabilization criterion.)
- Begin sampling by filling the sample containers slowly and continuously following the parameter order and guidance outlined in this instruction.
- Preserve the sample if necessary and verify that the pH is sufficient for the criteria.
- Check that a PTFE liner is present in the cap. Secure the cap tightly. Refer to C.4.3.3 for instructions on capping of VOA samples.

- Label the sample bottle with an appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately, along with the required trip blanks if analyzing for VOCs.

- Record the information in the field logbook and complete the chain-of-custody form and field sheets (see Instruction F-1, "Documentation," in Appendix F).

C.4.5 Field control samples requirements. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They include blanks, replicates, and/or background or upgradient samples. QA samples are replicates sent to a referee (QA) laboratory and analyzed to evaluate the contractor's laboratory performance. QC samples are blind replicates collected by the sampling team for analysis by the primary laboratory. A detailed discussion of field control samples is contained in Instruction G-2, Appendix G.

C.4.6 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the sample collection and handling as outlined previously and in Instruction F-1, Appendix F.

C.5 Sediment Sampling

C.5.1 Scope and application. This section presents guidelines for collecting representative sediment and sludge samples from surface water bodies. Sediment can be considered as any material that is submerged/saturated (at least temporarily) or suspended in any surface water body. This includes sludges, lake bottom sediments, perennial and intermittent stream sediments, and marine sediments. For discussion purposes, sampling devices are classified into the following categories according to applicability: surface sediments/shallow water (scoop and tube samplers), subsurface sediments/shallow water (hand auger/tube sampler, and hand-driven split-spoon sampler), surface sediments/deep water (Ponar, Ekman, and Smith-McIntyre samplers), and subsurface sediments/deep water (gravity and piston corer, vibratory coring device, and box core sampler).

C.5.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified in the HTRW technical planning process. The sampling strategy developed for a particular site will influence several project decisions, including, but not limited to, sampling locations, types of samples, sampling frequency, and sampling and analytical protocols. For instance, generation of an ecological risk assessment may require the need for colocated surface water and sediment samples. Sediment sampling must also determine whether the biota and/or underlying sediments are to be evaluated. Sampling strategies may be significantly influenced by such factors as physical site constraints, safety, and cost, to name a few. Additionally, the complexity, dynamic nature, and thin stratification of sediments may pose special challenges when determining appropriate sampling protocols. The technical planning process that results in the development of the sampling strategy is critical because of the difficulty in acquiring representative samples, the typically low-level contaminant action levels associated with sediments, and the problems associated with trace level cross-contamination. Successful investigations of hazardous waste sites are highly dependent on an effective sampling scheme. Development of a sampling scheme for purposes of characterizing a hazardous waste site should follow the fundamentals of the scientific approach. A successful sampling scheme requires a logical design to allow an evaluation of potential contaminants in relation to background conditions, vertical extent, horizontal extent, and mobility in various media. The USEPA is also an important source for guidance on sediment sampling. QA/QC guidance for sampling and analysis of sediments may be found in EPA/823/B-95/001.

C.5.2.1 Sampling locations. Sampling at hazardous waste sites is usually conducted in an attempt to discover contamination and to define its extent and variability. With such an objective, it is most logical to choose sample locations that will yield the most information about site conditions. During evaluation of a site, sampling can be conducted by random, systematic, or biased sampling. Biased samples are those collected at locations that were chosen based on historical information, knowledge about the behavior of the contaminant(s), and/or knowledge about the effects of the physical system on the fate of the contaminants. Random sampling depends on the theory of random chance probabilities to choose the most representative sample. Often biased and random sampling techniques can be used together to address an entire site thoroughly. Some samples may be biased to potentially contaminated areas (e.g., lagoons, former process or disposal areas) or potentially impacted areas (e.g., sediment downstream from a discharge pipe, or exposure area of a susceptible population). In areas less likely to be contaminated or areas with little available background information, random samples may be used to allow adequate assessment of the entire site. Due to the nature of the media, locations for collecting sediment samples are restricted to those within the water body under evaluation. Variations of locations for collecting sediment samples include sample location and depth. Sediment depositional patterns should be considered against the sample objectives when deciding the sediment sample locations. These patterns differ between standing and flowing bodies of water. Generally, for flowing water (e.g., streambeds or riverbeds), the depositional areas are normally found inside bends and downstream of islands or obstructions. Areas directly downstream of the joining of two streams should be avoided because the flows and sediments may not immediately mix. For standing water bodies,

the center of the mass or a discharge point should be sampled for sediments. As discussed in Section C.5.2, selection of sample locations should satisfy investigation objectives.

C.5.2.2 Types of samples. The type of sample should be designated when selecting a sampling method. Sediment samples can be discrete (grab) or composite. A discrete (grab) sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected at once and at one particular point in the sample matrix. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease. Composites are samples composed of more than one specific aliquot (discrete samples) collected at various sampling locations or depths. Analysis of this type of sample produces an average value and can in certain instances be used as an alternative to analyzing a number of individual grab samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix. When surficial sediments are sampled, the objectives of a project should determine whether any organic matter or strictly sediments should be sampled. The organic matter present may represent the primary contaminant source that aquatic species uptake.

C.5.2.3 Suggested samplers. Samplers for this medium are dictated significantly by project objectives of surficial versus subsurface samples and site constraints of the water depth. Each sampling technique presents various disadvantages and advantages for its application. For example, sample disturbance, sample volume, chemical/physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique. The advantages and disadvantages of each sampling technique are discussed in the following sections.

C.5.2.4 Sample frequency. Determination of the number of samples needed to characterize a site is also dependent upon sampling objectives and site-specific conditions. For example, if the objective of the event is to determine whether the site is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, the site is known to be contaminated and delineation of the contamination is the objective, a greater number of samples may be needed. In many cases, statistical considerations can be helpful in determining sampling strategy.

C.5.3 Sample preservation and handling. Many of the chemical constituents and physicochemical parameters that are to be measured or evaluated in investigation programs are not chemically stable; therefore, sample preservation is required. Appropriate preservation techniques for various parameters are specified in Appendix B. In addition, sample containers that should be used for each constituent or common set of parameters are specified in Appendix B. These preservation methods and sample containers are based on EPA/SW-846. Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2, "Packaging and Shipping Procedures," in Appendix F. Improper sample handling may alter the analytical results of the sample, causing the results to be invalid. When subsequent analysis allows, sediment samples should be collected using a clean stainless steel scoop, spoon, or trowel and placed into a clean stainless steel or other appropriate homogenization container. The sample should be mixed thoroughly to obtain a homogeneous, representative sample prior to placement into the sample container. Refer to Instruction E-2 of Appendix E for a discussion of homogenization procedures. When compositing of samples collected from different locations or depths is desired, all components of the composite sample are mixed in the homogenization container before the composite sample is placed in the sample container. Refer to Instruction E-3 of Appendix E for a discussion of compositing procedures. The sample should then be preserved in the field as specified in Appendix B. Because of the low analytical detection limits that are required for certain data uses, care must be taken when collecting the sample to avoid the loss or gain of any contaminants. For example, the samples packaged for volatile analysis should not be

homogenized or composited. They should be taken as described in Instruction E-4, "Collection, Handling, and Storage of Solid Samples for VOC Analysis" (Appendix E).

C.5.3.1 Sample containers. When metals are the analytes of interest, wide-mouth glass containers with PTFE-lined polypropylene caps should be used. When organics are the analytes of interest, glass bottles with PTFE-lined caps should be used. Refer to Appendix B or the specific analytical method to designate an acceptable container. Containers should be cleaned based on the analyte of interest. Instruction E-6, "Decontamination Procedures," Appendix E, contains additional information on appropriate glassware cleaning protocols. If precleaned bottles are used, the cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Refer to Appendix B for information on the required size and type of sample containers. Samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. A preferred collection order for some common parameters follows:

- VOA
- POC
- POX
- TOX
- TOC
- Extractable organics
- Total metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionuclides

C.5.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and to reduce sorption effects. Preservation methods for sediment samples are dependent on the type of analyses. For nonvolatile analyses, sample preservation is generally limited to refrigeration and/or protection from light. Volatile sediment sample preservation is based on whether the analysis is to be low-level or medium-level analysis. Refer to Instruction E-4 of Appendix E for details. The sampler should refer to Appendix B or the specific preservation method in EPA/SW-846 for the appropriate preservation technique.

C.5.3.3 Special handling for VOA samples. Samples to be analyzed for purgeable organic compounds should be acquired as described in Instruction E-4 of Appendix E, "Collection, Handling, and Storage of Solid Samples for VOC Analysis."

C.5.3.4 Special precautions for trace contaminant sampling. Contaminants can be detected in the parts per billion and/or parts per trillion ranges. Therefore, extreme care must be taken to prevent cross-contamination of these samples. The following general precautions should be taken when sampling:

C.5.3.4.1 A clean pair of new, disposable gloves should be worn each time a different location is sampled and gloves should be donned immediately prior to sampling.

C.5.3.4.2 To prevent cross-contamination between samples, it is suggested that the multiple VOA vials from each sampling location be sealed in separate smaller plastic bags when the sampled medium is suspected of containing high concentrations of volatile organics.

C.5.3.4.3 Sample containers filled with source or waste samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting and preserving, and activated carbon should be included in the bags to prevent cross-contamination.

C.5.3.4.4 If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants should be lined with new, clean, plastic bags.

C.5.3.4.5 If possible, one member of the field team should take all the notes, fill out sample tags, field sheets, etc., while the other members collect all of the samples.

C.5.3.4.6 Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.

C.5.3.4.7 Field personnel should take precautions to prevent contamination from sampling equipment. Some sediment samplers, particularly grab samplers, are constructed of metal and some may be electroplated or painted to prevent corrosion. PTFE-coated or stainless steel samplers are preferable. All samplers should be properly decontaminated before each use. When grab samplers are used, samples should be taken from the center of the mass of sediment retrieved by the sampler, avoiding material that has come in contact with the walls of the sampler. Liner materials or tubes for tube samplers should be selected to avoid sample contamination. For example, plastic liners or tape used to seal containers may be a source of contamination for organic compounds.

C.5.3.4.8 Adequate field control samples should be collected.

C.5.4 Sampling methods. Prior to sample collection, water body characteristics (size, depth, flow) should be recorded in the field logbook. Sampling should proceed from downstream locations to upstream locations so that disturbance from sampling does not affect sampling quality. Additionally, if surface water samples will be collected at the same locations as sediment samples, the water samples must be collected first. Factors that contribute to selection of a sampler include the width, depth, flow, and bed characteristics of the surface water body to be sampled, the volume of sample required, and whether the sample will be collected from the shore or a vessel. In collecting sediment samples from any source, care must be taken to

use appropriate sampling devices that minimize disturbance and sample washing as the sample is retrieved through the liquid column. Sediment fines may be carried out of the sample during collection and retrieval if the liquid above is flowing or deep. This may result in collection of a nonrepresentative sample due to the loss of contaminants associated with these fines. While a sediment sample is usually expected to be a solid matrix, the sampler should not place the sample in the sample bottle and then decant the excess liquid. If the sample is collected properly, any liquid in the bottle is representative of sediment conditions. As with surface water sampling, tidal influence on the water body should be determined, and the effect of the tide on the sediment sample collection should be detailed in the sampling plan. Consideration should be given to sampling at varied tidal stages. In addition, the stage of the tide at the time of sample collection should be recorded. In some instances, the dimensions of the water body dictate that a barge or boat must be used. The barge or boat should be positioned upstream (if there is flowing water) of the desired sample location. As the sampler is lowered it may be carried slightly downstream, depending upon the device used and the force of the flow. The device chosen for sample collection in this case will, again, depend upon the depth and flow of the liquid above the sample location and the bed characteristics of the surface water. The following are sampling instructions for the most common techniques for collecting sediment and sludge samples. For additional information see EM 1110-1-1906, EM 1110-2-5027, Plumb (1981), Mudrock and MacKnight (1991), and Spigolon (1993a, b). In addition, a comparison of the general characteristics of various sediment-sampling devices for chemical, physical, and biological studies can be found in ASTM D 4387, D 4823 and E 1391. The most appropriate device for a specific study depends on the study objectives, sampling conditions, parameters to be analyzed, and cost-effectiveness of the sampler. There are basically three types of devices used to collect sediment samples: dredges, grab samplers, and corers.

- A **dredge** is a vessel that is dragged across the bottom of the surface being sampled, collecting a composite of surface sediments and associated benthic fauna. This type of sampler is used primarily for collecting indigenous benthic fauna rather than samples for chemical analyses. Because the sample is mixed with the overlying water, no pore-water studies can be made of dredged samples. Additionally, because the walls of the dredge are typically nets, they act as a sieve and only the coarser material is trapped, resulting in the loss of fine sediments and water-soluble compounds. As noted earlier, this sample washing may potentially bias results to the low side. At best, results of dredge sampling are considered qualitative since it is difficult to determine the actual surface sampled by the dredge. For these reasons, dredge samplers are not addressed within this instruction.

- **Grab** samplers have jaws that close by a trigger mechanism upon impact with the bottom surface. Grab samplers offer the advantage of being able to collect a large amount of material in one sample, but they have the disadvantage of giving an unpredictable depth of penetration. Substantial contaminant variation with depth is unlikely in shallow channel areas without direct contamination inputs, in areas that have frequent ship traffic, or from sediments that are dredged at short intervals. In these situations, bottom sediments are frequently resuspended and mixed by ship scour and turbulence, effectively preventing stratification. In such cases, surface grab samples represent the mixed sediment column. Grab samplers are also appropriate for collecting surficial samples of reference or control sediments.

- **Core** samplers are basically tubes that are inserted into the sediment by various means to obtain a cylinder or box sample of material at known depths. Corers can be simple, hand-operated devices used by scuba divers, or they can be large, costly, motor-driven mechanisms that can collect samples from great depths. Corers are recommended whenever sampling to depth is required, or when the variation in contamination with depth is of concern. However, this type of data is necessary only if excavation of infrequently disturbed sediments below the mixed layer is planned. A few types of corers are a gravity corer, a piston corer, a vibra-corer, a split- spoon

core sampler, and a box core sampler. The choice of corer design depends on factors such as the objectives of the sampling program, sediment volumes required for testing, sediment characteristics, water depth, sediment depth, and currents or tides.

C.5.4.1 Surface sediments/shallow water: Scoop or trowel. Method Reference: ASTM D 5633.

C.5.4.1.1 Applicability. The scoop or trowel method is a very accurate procedure for collecting representative samples. This method can be used in many sampling situations but is limited to sampling exposed sediments or sediments in surface waters less than 150 mm (6 in.) deep, with nominal flow. The scoop or trowel sampler is not effective for sampling in waters more than 150 mm (6 in.) deep, or when flow causes a loss of fines from sample washing.

C.5.4.1.2 Method summary and equipment. The simplest, most direct method of collecting sediment samples is with the use of a stainless steel scoop or trowel (Figure C-10). A stainless steel scoop or trowel can be used to collect the sample, and a stainless steel bowl can be used to homogenize the sample when applicable to the subsequent analysis. The scoop or trowel should not be chrome-plated if metals are contaminants of concern.

C.5.4.1.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination.
- Sketch or photograph the sample area and note any recognizable features for future reference.
- Insert scoop or trowel into material and remove sample.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 (Appendix E) for additional information on the collection, handling, and storage of solid VOC samples.
- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, the sample is transferred to a stainless steel bowl for mixing.
- Repeat these steps as necessary to obtain sufficient sample volume.
- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 (Appendix E) as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.
- Check that a PTFE liner is present in cap. Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.

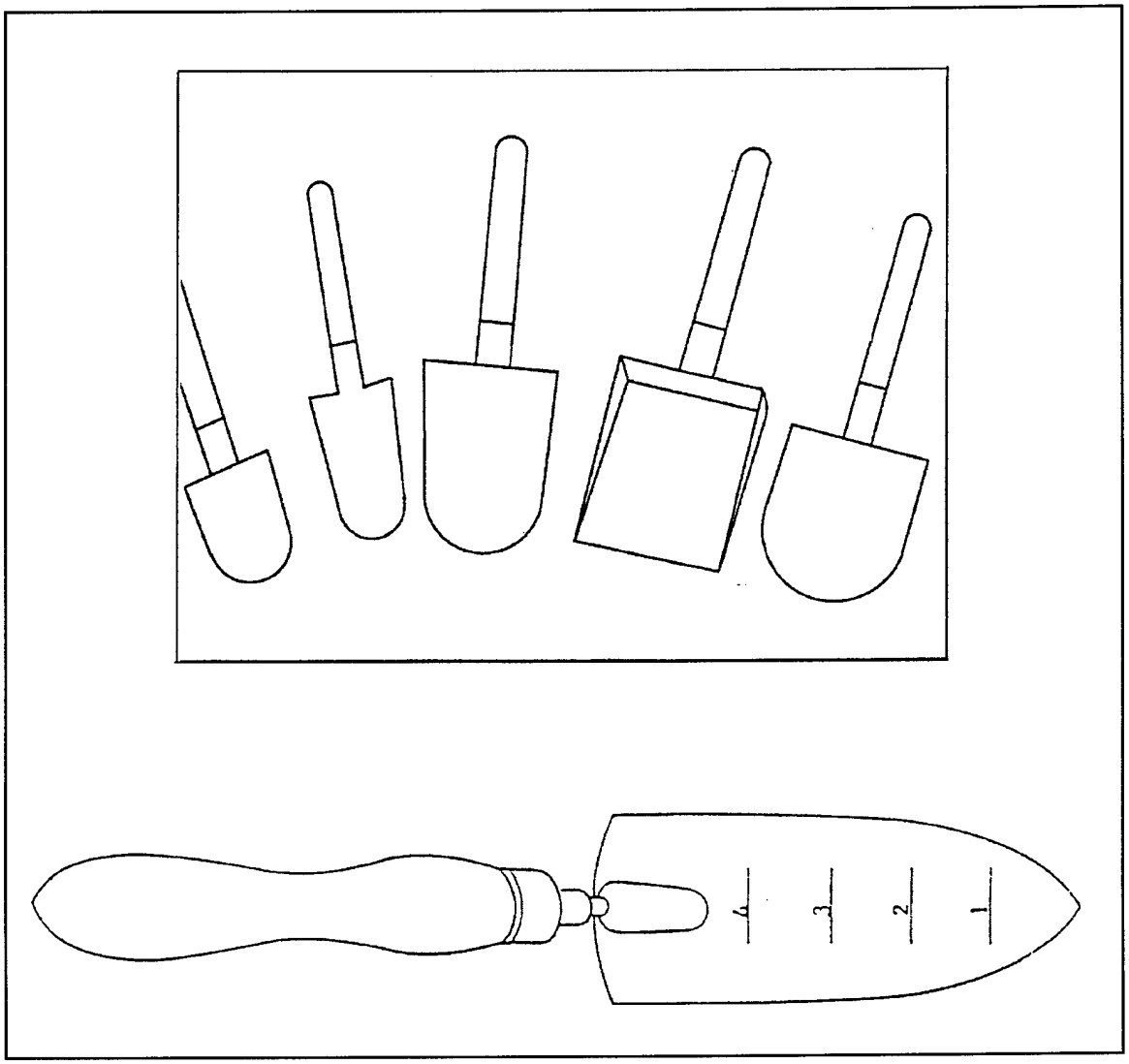


Figure C-10. Scoop trowel

- Complete all chain-of-custody documents and field sheets and record in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Decontaminate sampling equipment after use and between sample locations.

C.5.4.2 Surface sediments/shallow water: Tube sampler. Method References: ASTM D 4700 and D 4823.

C.5.4.2.1 Applicability. Equipment for the tube sampler is portable and easy to use (Figure C-11). Discrete sediment samples can be collected efficiently. Disadvantages of the tube sampler include its inability to collect sediment samples in water bodies greater than a few feet in depth and its inability to penetrate gravelly or rocky sediments.

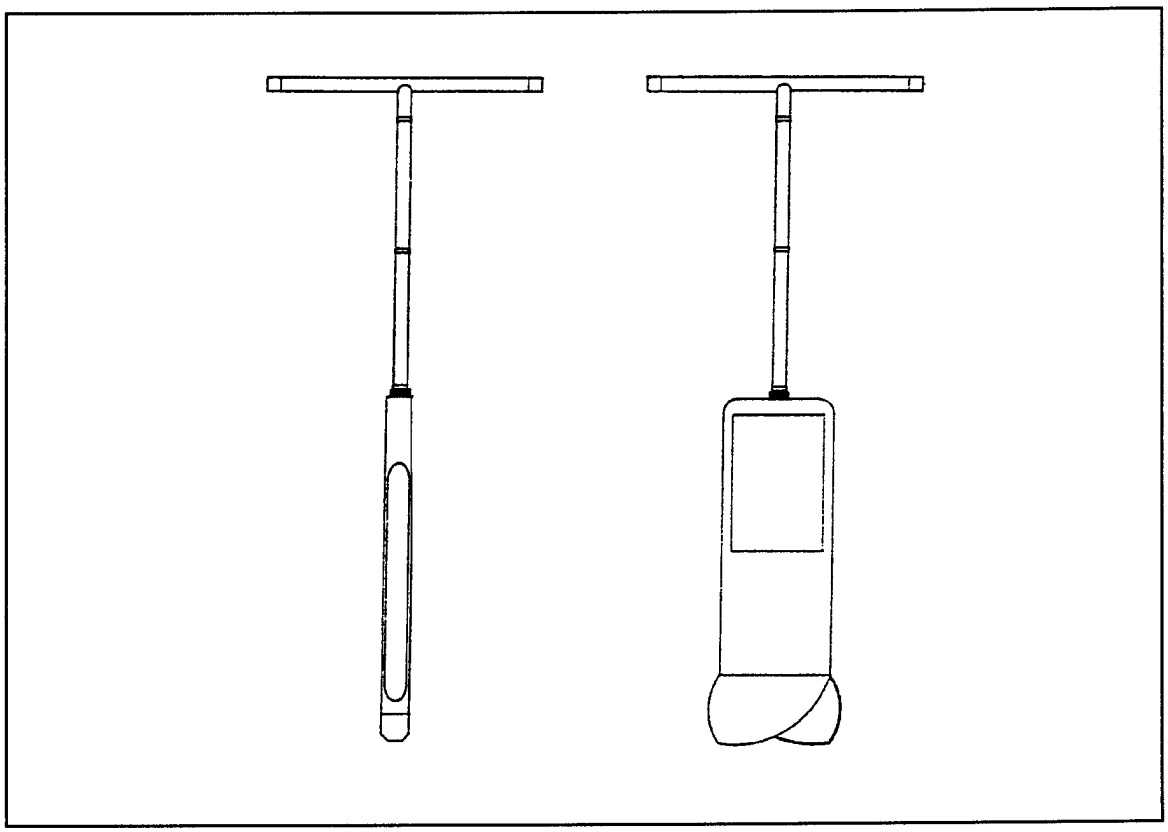


Figure C-11. Tube sampler and bucket auger

C.5.4.2.2 Method summary and equipment. Tube samplers are a simple and direct method for obtaining sediment samples. The tube sampler is forced into the sediment, then withdrawn and the sample is collected. In noncohesive soils, sample retention may be a problem.

C.5.4.2.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.
- Sketch or photograph the sample area and note any recognizable features for future reference.
- Gradually force tube sampler into sediment.
- Carefully retrieve the tube sampler.
- Remove sediment core from tube sampler and place core on a clean working surface.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 (Appendix E) for additional information on the collection, handling, and storage of solid VOC samples.

- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing.
- Repeat these steps as necessary to obtain sufficient sample volume.
- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate and collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.
- Complete all chain-of-custody documents and field sheets, and record in the field logbook (see Instruction F-1, Documentation).
- Decontaminate sampling equipment after use and between sample locations.

C.5.4.3 Subsurface sediments/shallow water: Hand auger and tube sampler. Method Reference: ASTM D 1452.

C.5.4.3.1 Applicability. Equipment for the hand auger and tube sampler is portable and easy to use (Figures C-11 and C-12). Discrete sediment samples can be collected efficiently. Disadvantages of the hand auger include its inability to collect sediment samples in water bodies greater than a few feet in depth and its inability to penetrate gravelly or rocky sediments. Also, hand augers may not be an effective method for augering into very soft sediments since the borehole may collapse prior to sampling.

C.5.4.3.2 Method summary and equipment. Hand augers are a simple and direct method for obtaining sediment samples. Although the maximum sampling depth for the hand auger is typically 1.5 m (5 ft), greater depths can be sampled depending on the sediment type. Hand augers come in various dimensions and various types. The bucket auger bit is used to bore a hole to the desired sampling depth and is then withdrawn. The auger tip is then replaced with the tube sampler, which is lowered into the borehole and forced into the sediment at the

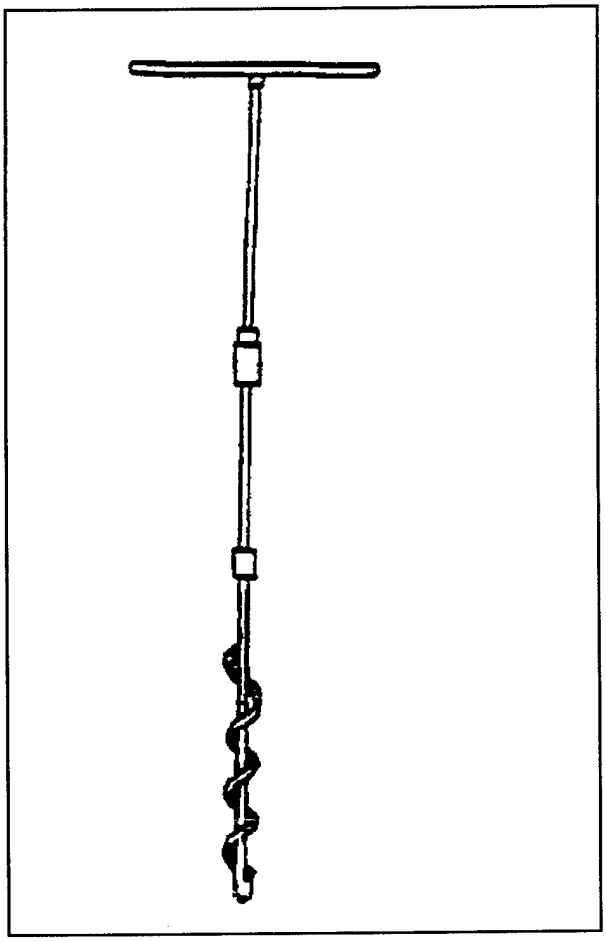


Figure C-12. Hand auger and tube sampler

desired depth. The corer is then withdrawn and the sample is collected. Potential problems encountered with this method include the collapsing or sloughing of the borehole after removal of the bucket auger. Relocating the borehole with the tube sampler may also be difficult if the water is turbid.

C.5.4.3.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform adjacent to the area to be sampled.
- Sketch or photograph the sample area and note any recognizable features for future reference.
- Attach the auger bit to a drill rod extension and attach the T-handle to the drill rod.
- Begin drilling. Periodically remove accumulated sediment to prevent accidentally brushing loose material into the borehole when removing the auger.
- After reaching the desired depth, slowly and carefully remove the auger from boring.
- Remove the auger tip from drill rods and replace with a precleaned or decontaminated thin-wall tube sampler. Install proper cutting tip.
- Carefully lower the tube sampler down borehole, and gradually force it into the sediment. Take care to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring because the vibrations may cause the boring wall to collapse.
- Carefully retrieve the tube sampler and unscrew drill rods.
- Remove cutting tip and remove core from device.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOC samples.
- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing.
- Repeat these steps as necessary to obtain sufficient sample volume.
- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.

- Complete all chain-of-custody documents and field sheets, and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Decontaminate sampling equipment after use and between sample locations.

C.5.4.4 Subsurface sediments/shallow water: Hand-driven split-spoon core sampler.

C.5.4.4.1 Applicability. The split-spoon core sampler may be used for obtaining sediment samples in cohesive and noncohesive sediments. Similar to the hand auger, the hand-driven split-spoon sampler can be used only in shallow water. However, because it is hammered into place, it can sometimes penetrate sediments that are too hard to sample with a hand auger.

C.5.4.4.2 Method summary and equipment. The split-spoon sampler is a 50.8-mm- (2-in.) diam, thick-walled, steel tube that is split lengthwise (Figure C-13). A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to the drill rods. For sediment sampling, the split-spoon sampler is usually attached to a short driving rod and driven into the sediment with a sledge hammer or slide hammer to obtain a sample.

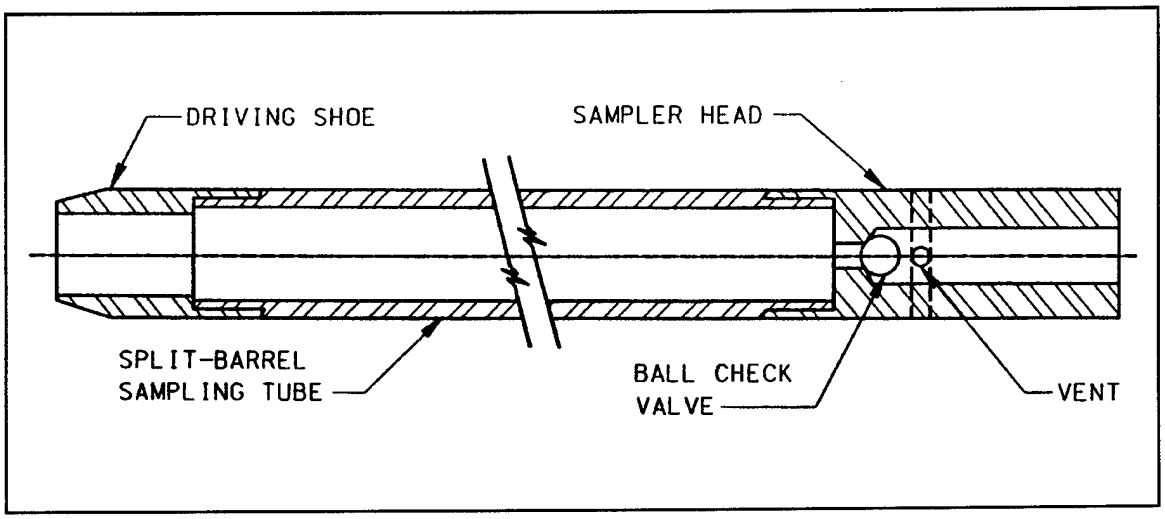


Figure C-13. Standard split-spoon sampler

C.5.4.4.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross- contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform upstream to the area to be sampled.

- Sketch or photograph the sample area and note any recognizable features for future reference.

- Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the heavier headpiece on top.

- Lower the sampler into position perpendicular to the material to be sampled.

- Drive the tube into the sediments with a sledge hammer. Do not drive past the bottom of the headpiece as this will result in compression of the sample.
- Withdraw the sampler and open by unscrewing drive shoe, head, and splitting barrel. If split samples are desired, use a decontaminated stainless steel knife to split the tube contents in half longitudinally.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOC samples.
- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing.
- Repeat these steps as necessary to obtain sufficient sample volume.
- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.
- Complete all chain-of-custody documents and field sheets, and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).
- Decontaminate sampling equipment after use and between sample locations.

C.5.4.5 Surface sediments/deep water: Ponar sampler. Method References: ASTM D 4342 and EPA/540/P-91/005, SOP #2016.

C.5.4.5.1 Applicability. Ponar samplers are capable of sampling most types of sludges and sediments from silts to granular materials. They are available in hand-operated sizes to winch- operated sizes. Ponars are relatively safe and easy to use, prevent escape of material with end plates, reduce shock waves, and have a combination of the advantages of other sampling devices. Ponar samplers are more applicable for a wide range of sediments and sludges because they penetrate deeper and seal better than spring-activated types (e.g., Ekman samplers). Penetration depths will usually not exceed several centimeters in sand. Greater penetration is possible in fine-grained material, up to the full depth of the sampler for soft sediments. Ponar samplers are not capable of collecting undisturbed samples. As a result, material in the first centimeter of sediment cannot be separated from the rest of the sample. Ponars can become buried in soft sediment.

C.5.4.5.2 Method summary and equipment. The Ponar sampler is a clamshell-type scoop activated by a counter-lever system (Figure C-14). The shell is opened, latched in place, and slowly lowered to the bottom. When tension is released on the lowering cable, the latch releases and the lifting action of the cable on the lever system closes the clamshell.

C.5.4.5.3 Sampling procedure. The sampling procedure is as follows:

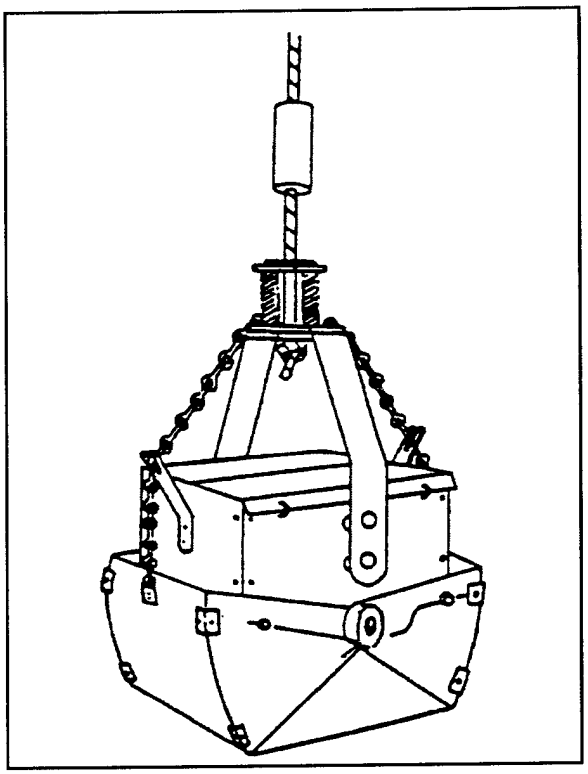


Figure C-14. Ponar sampler

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform upstream of the area to be sampled.
- Sketch or photograph the sample area and note any recognizable features for future reference.
- Attach a decontaminated Ponar to the necessary length of sample line. Solid braided 5-mm (3/16-in.) nylon line is usually of sufficient strength; however, 20-mm (3/4-in.) or greater nylon line allows for easier hand hoisting.
- Measure the depth to the top of the sediment with a weighted object.
- Mark the distance to the top of the sediment on the sample line with a proximity mark 1 m above the sediment. Record depth to top of sediment and depth of sediment penetration.
- Open sampler jaws until latched. From this point, support the sampler by its lift line, or the sampler will be tripped and the jaws will close.
- Tie the free end of sample line to fixed support to prevent accidental loss of sampler.
- Begin lowering the sampler until the proximity mark is reached.
- Lower the sampler at a slow rate of descent through last meter until contact is felt.
- Allow sample line to slack several centimeters. In strong currents, more slack may be necessary to release mechanism.
- Slowly raise Ponar grab sampler to clear surface.
- Drain free liquids through the screen of the sampler, being careful not to lose fine sediments.
- Place Ponar into a stainless steel or PTFE tray and open. Lift Ponar clear of the tray, and set aside for decontamination.

- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Samples should be taken from the center of the mass of sediment, avoiding material that has come in contact with the walls of the sampler. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.

- Repeat these steps until sufficient sample volume has been collected for remaining parameters.

- If compositing of different locations is desired, transfer additional discrete samples to a stainless steel bowl for mixing.
- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately.

- Complete all chain-of-custody documents and field sheets and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Decontaminate sampling equipment after use and between sample locations.

C.5.4.6 Surface sediments/deep water: Ekman grab sampler. Method References: ASTM D 4343 and EPA/540/P-91/005, SOP #2016.

C.5.4.6.1 Applicability. The Ekman sampler collects a standard size sample. The Ekman sampler is not useful in rough waters or if vegetation is on the bottom.

C.5.4.6.2 Method summary and equipment. The Ekman sampler (Figure C-15) is another clamshell-type grab sampler and works similarly to the Ponar sampler described previously. However, because the Ekman sampler is much lighter than the Ponar sampler, it is easier to handle and can even be attached to a pole for shallow applications. The Ekman sampler is unsuitable for sampling rocky or hard bottom surfaces.

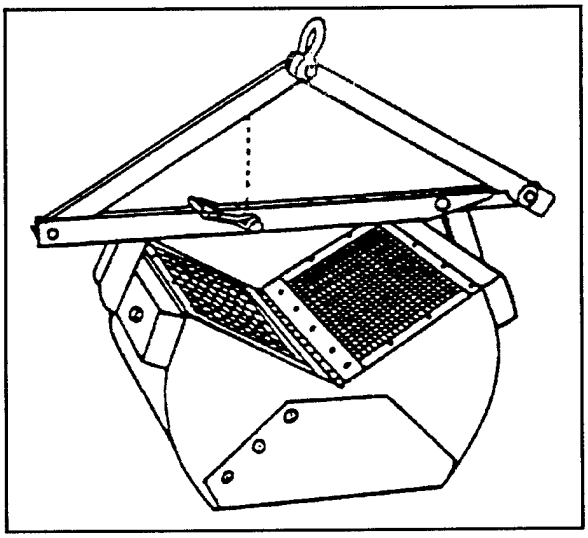


Figure C-15. Ekman sampler

C.5.4.6.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to

sampling location is restricted, locate a boat, barge, or other stable working platform upstream of the area to be sampled.

- Sketch or photograph the sample area and note any recognizable features for future reference.
- Attach a decontaminated Ekman sampler to the necessary length of sample line or in shallow waters to the end of a pole. Because the Ekman sampler is lightweight, solid braided 5-mm (3/16-in.) mylar line is sufficient.
- Measure the depth to the top of the sediment with a weighted object. Record the depth to top of sediment.
- Mark the distance to top of sediment on the sample line and add a proximity mark 1 m above the first mark so that the person taking the sample will know when he is approaching sediment.
- Open sampler jaws until latched. From this point, support the sampler by its lift line, or the sampler will be tripped and the jaws will close.
- If using a sample line, tie the free end of the sample line to fixed support to prevent accidental loss of sampler.
- Begin lowering the sampler until the proximity mark is reached.
- Lower the sampler at a slow rate of descent through the last meter until contact is felt.
- If using a sample line, place a messenger on the sample line and release, allowing the messenger to slide down to the sample line and activate the spring. Record the depth of sediment penetration by the sampler.
- Slowly raise Ekman grab sampler to clear surface.
- Drain free liquids through the screen of the sampler, being careful not to lose fine sediments.
- Place Ekman sampler into a stainless steel or PTFE tray and open. Lift Ekman sampler clear of the tray and set aside for decontamination.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Samples should be taken from the center of the mass of sediment, avoiding material that has come in contact with the walls of the sampler. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.
- Repeat these steps until sufficient sample volume has been collected for remaining parameters.
- If compositing of different locations is desired, transfer additional discrete samples to stainless steel bowl for mixing.

- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately.

- Complete all chain-of-custody documents and field sheets and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Decontaminate sampling equipment after use and between sample locations.

C.5.4.7 Surface sediment/deep water: Smith-McIntyre grab sampler. Method Reference: ASTM D 4344.

C.5.4.7.1 Applicability. The Smith-McIntyre grab sampler can be used in rough water because of its large and heavy construction. It reduces premature tripping and can be used in depths up to 1,050 m (3,500 ft). The flange on the jaws reduces material loss. It is good for sampling all sediment types. However, because of its large and heavy construction, the Smith-McIntyre sampler is cumbersome to operate.

C.5.4.7.2 Method summary and equipment. The Smith-McIntyre grab sampler (Figure C-16) is also a type of clam-shell-style grab sampler and works similarly to the Ponar sampler described previously.

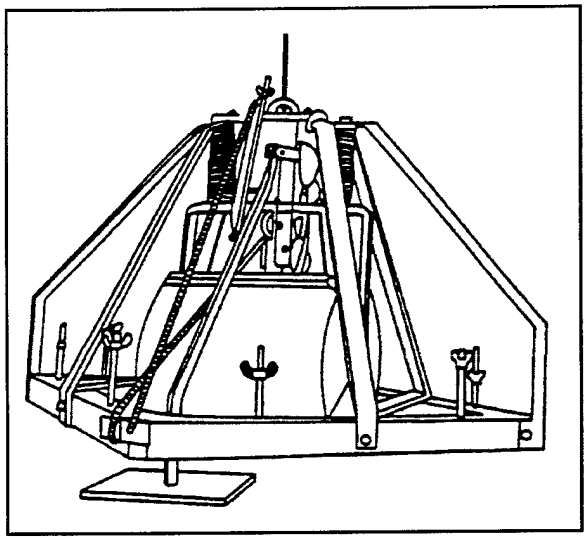


Figure C-16. Smith-McIntyre sampler

C.5.4.7.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the deck of a boat or barge to keep sampling equipment decontaminated and to prevent cross-contamination.

- Sketch or photograph the sample area and note any recognizable features for future reference.

- Attach a decontaminated Smith-McIntyre sampler to the necessary length of sample line. Because the Smith-McIntyre sampler is large and heavy, a winch should be used for hoisting and lowering the sampler.

- Measure the depth to the top of the sediment with a weighted object.

- Mark the distance to top of sediment on the sample line with a proximity mark 2.5 cm (1 in.) above the sediment. Record depth to top of sediment and depth of sediment penetration.

- Open sampler jaws until latched. From this point, support the sampler by its lift line, or the sampler will be tripped and the jaws will close.
- If using a sample line, tie the free end of sample line to fixed support to prevent accidental loss of sampler.
- Begin lowering the sampler until the proximity mark is reached.
- Lower the sampler at a slow rate of descent through last meter until contact is felt.
- Allow sample line to slack several centimeters. In strong currents, more slack may be necessary to release mechanism.
- Slowly raise Smith-McIntyre grab sampler to clear surface.
- Drain free liquids through the screen of the sampler, being careful not to lose fine sediments.
- Place Smith-McIntyre sampler into a stainless steel or PTFE tray and open. Lift Smith-McIntyre sampler clear of the tray and set aside for decontamination.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Samples should be taken from the center of the mass of sediment, avoiding material that has come in contact with the walls of the sampler. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.
- Repeat these steps until sufficient sample volume has been collected for remaining parameters.
- If compositing of different locations is desired, transfer additional discrete samples to stainless steel bowl for mixing.
- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.
- Complete all chain-of-custody documents and field sheets and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).
- Decontaminate sampling equipment after use and between sample locations.

C.5.4.8 Subsurface sediments/deep water: Gravity and piston corers. Method References: EM 1110-1-1906, ASTM D 4823, Mudroch and MacKnight (1991), and American Public Health Association (1995).

C.5.4.8.1 Applicability. Gravity corers are capable of collecting samples of most sludges and sediments. They collect essentially undisturbed samples that represent the profile of strata that may develop in sediments and sludges during variations in the deposition process. The gravity corer may be limited to cores of 1 to 2 m (3 to 6.5 ft) in depth, depending on sediment grain size, degree of sediment compaction, and velocity of the drop. Because gravity corers can compact the sample and distort the vertical profile, a vibratory corer is recommended to minimize sample compaction and when vertical stratification in a core sample is of interest. If the samples will not be sectioned prior to analysis and compaction is not a problem, the gravity (free-fall) corers may be the simplest alternative. The piston corer is similar to a gravity corer but also has a piston inside the tube that remains stationary during sediment penetration and creates a vacuum that helps pull the sampler into the sediment. The piston corer uses both gravity and hydrostatic pressure. Refer to Figure C-20 within Instruction C-6 for a schematic of a piston corer (sampler). As the cutting edge of the corer penetrates the sediments, an internal piston remains at the level of the sediment/water interface, preventing sediment compression and overcoming internal friction.

C.5.4.8.2 Method summary and equipment. The gravity corer uses weights attached to the head of the sampling tube to push the tube into the sediment. It is a metal tube with a replaceable tapered nosepiece on the bottom and a ball or other type of check valve on the top. The check valve allows water to pass through the corer on descent but prevents a washout during recovery. The tapered nosepiece facilitates cutting and reduces core disturbance during penetration. Most corers are constructed of brass or steel, and many can accept plastic liners and additional weights.

C.5.4.8.3 Sampling procedure. The sampling procedure is as follows:

- Spread new plastic sheeting on the ground at each sampling location to keep sampling equipment decontaminated and to prevent cross-contamination. If access to sampling location is restricted, locate a boat, barge, or other stable working platform upstream of the area to be sampled.
- Sketch or photograph the sample area and note any recognizable features for future reference.
- Attach a decontaminated corer to the required length of sample line. Solid braided 5-mm (3/16-in.) nylon line is typically sufficient; 20-mm (3/4-in.) nylon, however, is easier to grab during hand hoisting.
- Secure the free end of the line to a fixed support to prevent accidental loss of the corer.
- Allow corer to free fall through liquid to bottom.
- Retrieve corer with a smooth, continuous lifting motion. Do not bump corer as this may result in some sample loss.
- Remove nosepiece from corer and slide sample out of corer into stainless steel or PTFE pan.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.
- Repeat these steps until sufficient sample volume has been collected for remaining parameters.
- If compositing of different locations is desired, transfer additional discrete samples to stainless steel bowl for mixing.

- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, and transfer into an appropriate sample bottle.

- Check that a liner is present in cap. Secure the cap tightly.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately.

- Complete all chain-of-custody documents and field sheets and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Thoroughly decontaminate the gravity corer after each use.

C.5.4.9 Subsurface sediments/deep water: Vibratory coring device.

C.5.4.9.1 Applicability. Vibratory corers are capable of collecting samples of most soils, sediments, and sludges. For penetration greater than 2 m (6.5 ft), a vibratory corer is generally preferred.

C.5.4.9.2 Method summary and equipment. The vibratory system consists of a tripod that supports a core tube. An external power source is necessary to drive a top head and cause vibrations. The vibratory motion causes the soil sediments to become fluidized and the core tube to slip through the soil or sediment. It is capable of obtaining 3- to 7-m cores in a wide range of sediment types by vibrating a large diameter core barrel through the sediment column with little compaction. For additional information, see EM 1110-1-1906, Finkelstein and Prins (1981), Meisburger and Williams (1981), U.S. Army Engineer Waterways Experiment Station (1982), and Smith (1993).

C.5.4.9.3 Sampling procedure. The sampling procedure is as follows:

- Locate a boat, barge, or other stable working platform over the area to be sampled.

- Sketch or photograph the sample area and note any recognizable features for future reference.

- Assemble a decontaminated vibratory corer and connect an external power source (i.e., air compressor).

- Attach decontaminated corer to the required length of sample line to reach the top of the soil or sediment.

- Lower the corer down to the top of sediments and begin vibratory coring until the core tube has fully penetrated.

- Carefully retrieve the core tube and remove the core liner.

- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.

- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, transfer into an appropriate sample bottle, secure cap tightly, and put the container on ice.

- Complete all chain-of-custody documents and field sheets and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Thoroughly decontaminate the vibratory corer after each use.

C.5.4.10 Subsurface sediments/deep water: Box core sampler.

C.5.4.10.1 Applicability. The corer that disturbs the sediments the least is a box corer. One advantage of the box corer is its ability to collect a large amount of sample with the center of the sample virtually undisturbed. Box corers are not generally recommended for use in sandy sediments since they have difficulty retaining the sample upon withdrawal.

C.5.4.10.2 Method summary and equipment. The box corer is a large box-shaped sampler that is deployed inside a frame. After the frame is brought to rest on the bottom, heavy weights lower the open-ended box into the sediment. A bottom door then swings shut upon retrieval to prevent sample loss.

C.5.4.10.3 Sampling procedure. The sampling procedure is as follows:

- Locate a boat, barge, or other stable working platform over the area to be sampled.

- Sketch or photograph the sample area and note any recognizable features for future reference.

- Assemble decontaminated box corer into the sample frame.

- Attach decontaminated box corer and frame to the required length of sample line, cable, or rope to reach the top of the soil or sediment.

- Secure the free end of the line to a fixed support to prevent accidental loss of the corer, if applicable.

- Lower the box corer and frame down to the sediments. Weights will force the box corer into the sediments for sample collection.

- Carefully retrieve the box corer with a smooth, continuous lifting motion. The box bottom will swing shut upon retrieval.

- Open the bottom lid to remove sample out of corer into stainless steel or PTFE pan.

- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.

- Repeat these steps until sufficient sample volume has been collected for remaining parameters.

- If compositing of different locations is desired, transfer additional discrete samples to a stainless steel bowl for mixing.

- Thoroughly mix remaining sample as outlined in Instructions E-2 and E-3 as appropriate, collect suitable aliquots with a stainless steel laboratory spoon or equivalent, transfer into an appropriate sample bottle.

- Check that a liner is present in cap. Secure the cap tightly.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place filled sample containers on ice immediately.

- Complete all chain-of-custody documents and field sheets and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F).

- Thoroughly decontaminate the box corer after each use.

C.5.5 Decontamination procedures. All equipment that will enter the sediment must be decontaminated as described in Instruction E-6 (Appendix E). Sampling equipment should be placed in plastic bags until immediately prior to use. Additional sampling devices may be needed onsite to ensure an adequate drying time.

C.5.6 Field control sample requirements. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They include blanks, replicates, and/or background samples. QA samples are replicates sent to a referee (QA) laboratory and analyzed to evaluate the contractor's laboratory performance. QC samples are blind replicates collected by the sampling team for analysis by the primary laboratory. A detailed discussion of field control samples is contained in Instruction G-2 (Appendix G).

C.5.7 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site setup, sample collection, and handling as outlined previously and in Instruction F-1, Appendix F.

C.5.8 Analytical techniques for sediment samples. Techniques for chemical analysis of sediments have some inherent limitations. Interferences encountered as part of the sediment matrix, particularly in samples from heavily contaminated areas, may limit the ability of a method to detect or quantify some analytes at the required levels. Sediment analyses usually require lower reporting limits than for soils because regulatory or effects levels are, in general, lower in sediments than in soil. These lower reporting levels may require a change in calibration of analytical instrumentation using lower concentration standards. Lower spiking concentrations will probably be required to better represent the recoveries in low concentration samples. For many metals analyses in marine/estuarine areas, the concentration of salt may

be much greater than the analyte of interest and can cause unacceptable interference in certain analytical techniques. For this reason and the need for lower detection limits, analytical methods for soils may need to be modified or different methods selected. The analyses for semivolatile and VOCs may also need to be modified to achieve lower detection limits and cleanup of this complex matrix. Extensive cleanup is required because of the likely presence of biological macromolecules, which accumulate in sediments; sulfur from sediments with low or no oxygen; and oil and/or grease in the sediment. In addition to all of the previously stated differences between soil and sediment analyses, sediments may require the analysis for other constituents. For example, knowing the concentration of TOC is important for evaluating the bioavailability of neutral organics (i.e. PAH's. PCB's, dioxin, and chlorinated pesticides). In areas with marinas or high ship and boat use, it may be necessary to analyze the sediment for tri-butyl tin (TBT), a highly toxic and persistent component of antifouling paint.

C.6 Soil Sampling

C.6.1 Scope and application. This instruction presents guidance for collecting representative soil samples. Soil sampling can be classified into two primary types: surface and subsurface. Instructions for sampling surface and subsurface soils by the following techniques are included in this instruction: spade and scoop, hand auger and tube sampler, split-spoon sampler, ring-lined barrel sampler, thin-walled (Shelby) tube, continuous tube sampler, piston sampler, core barrel sampler, direct push, and site characterization and analysis penetrometer system (SCAPS). EM 1110-1-1906 also addresses these and other types of geotechnical soil sampling, which may be adapted for environmental purposes.

C.6.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified in the HTRW technical planning process. The sampling strategy developed for a particular site will influence several project decisions, including, but not limited to, sampling locations, sample depths, types of samples, sampling frequency, and sampling and analytical protocols. Clarification of key definitions may need to be addressed. For instance, surface soils should be defined in terms of depth requirements. For depending on the data use, surface soils may be defined as the 0-5 cm (0-2 inch), or the 0-15 cm (0-6 inch) depth interval. Sampling strategies may also be significantly influenced by such factors as matrix and contaminant characteristics, physical site constraints, safety, and cost, to name a few. Soil sampling pose a variety of challenges due to the natural variability of the media, the lack of understanding of contaminant migration through the vadose zone, and the logistical problems of sampling at increased depths. Additionally, the particle size distribution of the soils must be evaluated against the scale of the contaminant. Contaminants present on a macro-scale (i.e., lead shot, solid contaminant material or chunks) is more susceptible to bias during sampling procedures, than contaminants found on a molecular scale. The technical planning process that results in the development of the sampling strategy is critical because of these difficulties in acquiring representative samples, the reduction of contaminant action levels, and the problems associated with trace level cross-contamination. Development of a sampling scheme to characterize a hazardous waste site should follow the fundamentals of scientific approach. A successful sampling scheme requires a logical design to allow an evaluation of potential contaminants in relation to background conditions, vertical extent, horizontal extent, and mobility in various media.

C.6.2.1 Sampling locations. Sampling at hazardous waste sites is usually conducted in an attempt to discover contamination and to define its extent and variability. With such an objective, it is most logical to choose sample locations that will yield the most information about site conditions. During evaluation of a site, sampling can be conducted by random, systematic, or biased sampling. Biased samples are those collected at locations that were chosen based on historical information, knowledge about the behavior of the contaminant(s), and/or knowledge about the effects of the physical system on the fate of the contaminant. Random sampling depends on the theory of random chance probabilities to choose the most representative sample. Often, biased and random sampling techniques can be used together to thoroughly address an entire site. Some samples may be biased to potentially contaminated areas (e.g., stained soil, former process or disposal areas) or potentially impacted areas (e.g., areas of stressed vegetation). In areas less likely to be contaminated or areas with little available background information, random samples may be used to allow adequate assessment of the entire site. Because of the nature of the media, soil samples can vary considerably across a site. Physical properties of the soil, including grain size and cohesiveness, may limit the depth from which samples can be collected and the method required to collect them. In most soils, hand-powered equipment can be used only to a depth of approximately 120 to 150 cm (4 to 5 ft). At greater depths, soil sampling is normally performed with a drill rig or other mechanically driven device.

C.6.2.2 Types of samples. The type of sample should be designated when selecting a sampling method. Application techniques for sample methods include discrete (grab) or composite samples. A

discrete (grab) sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected immediately and at one particular point in the sample matrix. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease. Composites are samples composed of two or more specific aliquots (discrete samples) collected at various sampling locations or depths. Analysis of this type of sample produces an average value and can, in certain instances, be used as an alternative to analyzing a number of individual grab samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix. Samples can be collected manually if soil conditions are favorable and the desired depth of sampling is not too great. Manual sampling involves minimal initial cost, and the method is well suited to a relatively small or specific number of samples. At depths greater than 120 to 150 cm (4 to 5 ft), manual sampling will probably not be possible, and a mechanically driven drilling device will be required. Depending on the sampling requirements of the site, the use of a mechanical drilling device can substantially increase the cost of a sampling investigation. However, it is usually the only method available to obtain soil samples at depths greater than a few feet. There are a great variety of mechanical drilling devices available for soil sampling. Discussions concerning the use of mechanical drilling devices will be limited to the actual tools used to collect the soil samples.

C.6.2.3 Suggested samplers. Each sampling technique presents various disadvantages and advantages for its application. For example, sample disturbance, sample volume, chemical/physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination varies from technique to technique. Subsurface soil conditions themselves will restrict the application of certain samplers. For example, the thin-walled tube sampler is not applicable for sampling sands. Discussions of the advantages and disadvantages of each sampling technique are presented in the following sections.

C.6.2.4 Sample frequency. Determination of the number of samples needed to characterize a site also depends on the objectives and the site-specific conditions. For example, if the objective of the event is to determine whether the site is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, the site is known to be contaminated and delineation of the contamination is the objective, a greater number of samples may be needed. In many cases statistical considerations can be helpful in determining sampling strategy. Additional guidance concerning sample frequency can be found in other U.S. Army Corps of Engineers (USACE) guidance and in ASTM D 5911, D5254, and D5792.

C.6.3 Sample preservation and handling. Many of the chemical constituents and physicochemical parameters that are to be measured or evaluated in soil investigation programs are not chemically stable; therefore, sample preservation is required. Appropriate preservation techniques for various parameters are specified in Appendix B. In addition, sample containers that the sampler should use for each constituent or common set of parameters are specified in Appendix B. These preservation methods and sample containers are based on EPA/SW-846. Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2, "Packaging and Shipping Procedures," Appendix F. Improper sample handling may alter the analytical results of the sample, causing the results to be invalid. When subsequent analysis allows, soil samples should be collected using a clean stainless steel scoop, spoon, or trowel and placed into clean stainless steel or other appropriate homogenization containers. Homogenization procedures are discussed in Instruction E-2, Appendix E. The sample should be mixed thoroughly to obtain a homogeneous, representative sample prior to placement into the sample container. When composited samples from different locations or depths are desired, all components of the composite sample are mixed in the homogenization container before the composite is placed in the sample container. Compositing procedures are discussed in Instruction E-3, Appendix E. The sample should then be preserved in the field as specified in Appendix B. Because of the low analytical detection limits that are required for certain data

uses, care must be taken when collecting the sample to avoid the loss or gain of any contaminants. For example, the samples packaged for volatile analysis should not be homogenized or composited. They should be handled in a manner as outlined in Instruction E-4, "Collection, Handling, and Storage of Solid Samples for VOC Analysis" in order to minimize contaminant loss through agitation/ volatilization.

C.6.3.1 Sample containers. When metals are the analytes of interest, wide-mouth glass jar containers with PTFE-lined polypropylene caps should be used. When organics are the analytes of interest, glass bottles with PTFE-lined caps should be used. Refer to Appendix B or the specific analytical method to designate an acceptable container. Containers should be cleaned based on the analyte of interest. Instruction E-6, "Decontamination Procedures," contains additional information on appropriate glassware cleaning protocols. If precleaned bottles are used, the cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Refer to Appendix B or the specific analytical method for information on the required size and type of sample containers. Samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. A preferred collection order for some common parameters follows:

- VOA
- POC
- POX
- TOX
- TOC
- Extractable organics
- Total metals
- Cyanide
- Sulfate and chloride
- Nitrate and ammonia
- Radionuclides

C.6.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and to reduce sorption effects. Preservation methods for soil samples are dependant on the type of analyses. For nonvolatile analysis, sample preservation is generally limited to refrigeration and/or protection from light. Volatile soil sample preservation is based on whether analysis is to be low-level or medium-level analysis. Refer to Instruction E-4, Appendix E, for details. The sampler should refer to Appendix B or the specific preservation method in EPA/SW-846 for the appropriate preservation technique.

C.6.3.3 Special handling for VOA samples. Samples to be analyzed for purgeable organic compounds should be acquired as described in Instruction E-4 of Appendix E, "Collection, Handling, and Storage of Solid Samples for VOC Analysis."

C.6.3.4 Special precautions for trace contaminant sampling. Contaminants can be detected in the parts per billion and/or parts per trillion ranges. Therefore, extreme care must be taken to prevent cross-contamination of these samples. The following general precautions should be taken when sampling:

- A clean pair of new, disposable gloves should be worn each time a different location is sampled and gloves should be donned immediately prior to sampling.
- All work should be conducted on a clean surface, such as a stainless steel table.
- To prevent cross-contamination between samples, it is suggested that the multiple vials from each sampling location be sealed in separate smaller plastic bags when the sampled medium is suspected of containing high concentrations of volatile organics.
- Sample containers filled with source or waste samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting and preserving, and activated carbon should be included in the bags to prevent cross-contamination.
- If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants should be lined with new, clean, plastic bags.
- If possible, one member of the field team should take all the notes, fill out sample tags, field sheets, etc., while the other members collect all of the samples. This is especially important when subjective decisions such as soil type and descriptions are being made.
- Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.
- Field personnel should take precautions to prevent contamination from sampling equipment. Most soil samplers are constructed of metal. Stainless steel samplers are preferable. All samplers should be properly decontaminated and inspected for visible signs of deterioration before each use. Samples should be taken from the center of the mass of soil retrieved by the sampler, avoiding material that has come in contact with the walls of the sampler. Liner materials or tubes for tube samplers may be selected to avoid sample contamination. For example, plastic liners or tape used to seal containers may be a source of contamination for organic compounds.
- Adequate field control samples should be collected.

C.6.4 Sampling methods. Presented in the following sections are sampling instructions for the most common techniques of collecting soil samples. Prior to sample collection, the soil sampling location and characteristics (soil type, depth) should be recorded in the field logbook. Selection of soil sampling equipment is usually based on the depth of the samples. Manual techniques are usually selected for surface or shallow subsurface soil sampling. At greater depths, mechanically driven equipment is usually required to overcome torque induced by soil resistance and depth. Additional information on collecting soil samples is presented in EPA/540/4-91/001, EPA/625/R-93/003A, ASTM D 4700, ASTM D 6169, and D 5730. ASTM Standards for specific applications of waste management include D 4547 and C 998.

C.6.4.1 Spade and scoop. Method Reference: ASTM D 5633.

C.6.4.1.1 Applicability. The spade and scoop method is a very accurate, representative method for collecting surface and shallow subsurface soil samples. This method is usually limited to soil depths less than 30 cm (1 ft).

C.6.4.1.2 Method summary and equipment. The simplest, most direct method of collecting surface soil samples is to use a spade and stainless steel scoop (Figure C-10 in Instruction C-5). A typical garden spade can be used to remove the top cover of soil to the required depth, but the smaller stainless steel scoop should be used to collect the sample. When a garden spade is used, the spade should be decontaminated before use; and if the spade is driven into the soil with the sampler's field boot, the boot should be covered with a clean disposable overboot. Typical garden-type scoops are many times plated with chrome or other metals and would, therefore, be inappropriate for sampling when analyzing for metals.

C.6.4.1.3 Sampling procedure. The guidance provided for use in shallow sediment collection, Section C.5.4.1.3, is appropriate for surficial soils collection with a spade or scoop.

C.6.4.2 Hand auger and tube sampler. Method Reference: ASTM D 1452 and D 4700.

C.6.4.2.1 Applicability. Equipment for the hand auger is portable and easy to use. Discrete subsurface soil samples can be collected efficiently without the use of a drill rig. Disadvantages of the hand auger include its limited sampling depth. The tube sampler may not penetrate gravelly or rocky soils.

C.6.4.2.2 Method summary and equipment. Hand augers are the simplest and most direct method for sampling subsurface soil samples (Figure C-11 and Figure C-12). Although the maximum sampling depth for the hand auger is typically 1.5 m (5 ft), greater depths can be sampled depending on the soil type. Hand augers come in various diameters and various types. The auger bit is used to bore a hole to the desired sampling depth and then withdrawn. The auger tip is then replaced with the tube corer, lowered into the borehole, and forced into the soil at the completion depth. The corer is then withdrawn and the sample is collected.

C.6.4.2.3 Sampling procedure. The guidance provided for use in sediment collection, Section C.5.4.3.3, is appropriate for soils collection with a hand auger and tube sampler.

C.6.4.3 Split-spoon sampler. Method Reference: ASTM D 1586.

C.6.4.3.1 Applicability. The split-spoon sampler is used for sampling subsurface soil in cohesive- and noncohesive-type soils. It is used extensively for collecting subsurface soil samples for chemical analysis. The split-spoon sampler will require a drill rig and crew for collecting samples greater than 1.5 m (5 ft).

C.6.4.3.2 Method summary and equipment. The split-spoon sampler is typically a 5- to 11.5-cm- (2- to 4-1/2-in.-) diam., thick-walled, steel tube that is split lengthwise (Figure C-13). If a 5-cm- (2-in.-) diam. split-spoon sampler is used, then standard penetration tests can be taken to determine the density of the soil. A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to the drill rods. When a boring is advanced to the point that a sample is to be taken, drill tools are removed and the sampler is lowered into the hole on the bottom of the drill rods. The sampler is driven into the ground in accordance with the standard penetration test (Appendix B of EM 1110-1-1906). Recently, the U.S. Army Engineer District, Mobile, has remanufactured the connection between the split-spoon sampler

and the drill rods headpiece from threads to a quick connect/release mechanism. The sampler is referred to as the Sanford quick-connect split- spoon sampler, named after its designer, Johnny Sanford. The Sanford split-spoon sampler (Figure C-17) is manufactured with one-half of the split spoon permanently attached to the sampler head, the other half sliding up into the head, and the driving shoe threaded onto both halves completing the assembly. By eliminating the need to screw and unscrew the upper end of the split tube, there is less disturbance to the soil core and a significant amount of time is saved.

C.6.4.3.3 Sampling procedure. The sampling procedure is as follows:

- Place plastic sheeting on the ground around the sampling location to prevent cross-contamination.
- Assemble the sampler by aligning both sides of the barrel and then screwing the drive shoe on the bottom and the sampler head on top. The Sanford quick-connect split-spoon sampler is assembled as noted in the preceding section.
- Place the sampler in a perpendicular position on the material to be sampled.
- Drive the tube, using a sledge hammer or drill rig if available. Do not drive past the bottom of the headpiece because this will result in compression of the sample.
- Record the length of the tube that penetrated the material being sampled and the number of blows required to obtain this depth. Typically, the number of blows per 15 cm (6 in.) of depth is recorded.
- Withdraw the sampler, disconnect from the drill rods (if necessary), and open it by unscrewing the drive shoe and head and splitting the barrel. Record appropriate information for soil core logging per EM 1110-1-4000 (i.e., soil depth, type, and classification).
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. If split samples are desired, a decontaminated stainless steel knife should be used to split the tube contents in half longitudinally. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.
- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing. Refer to Instructions E-2 and E-3, Appendix E, respectively, for procedures to follow for these techniques.
- Transfer sample into an appropriate sample bottle with a stainless steel laboratory spoon or equivalent.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to label the bottle carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.

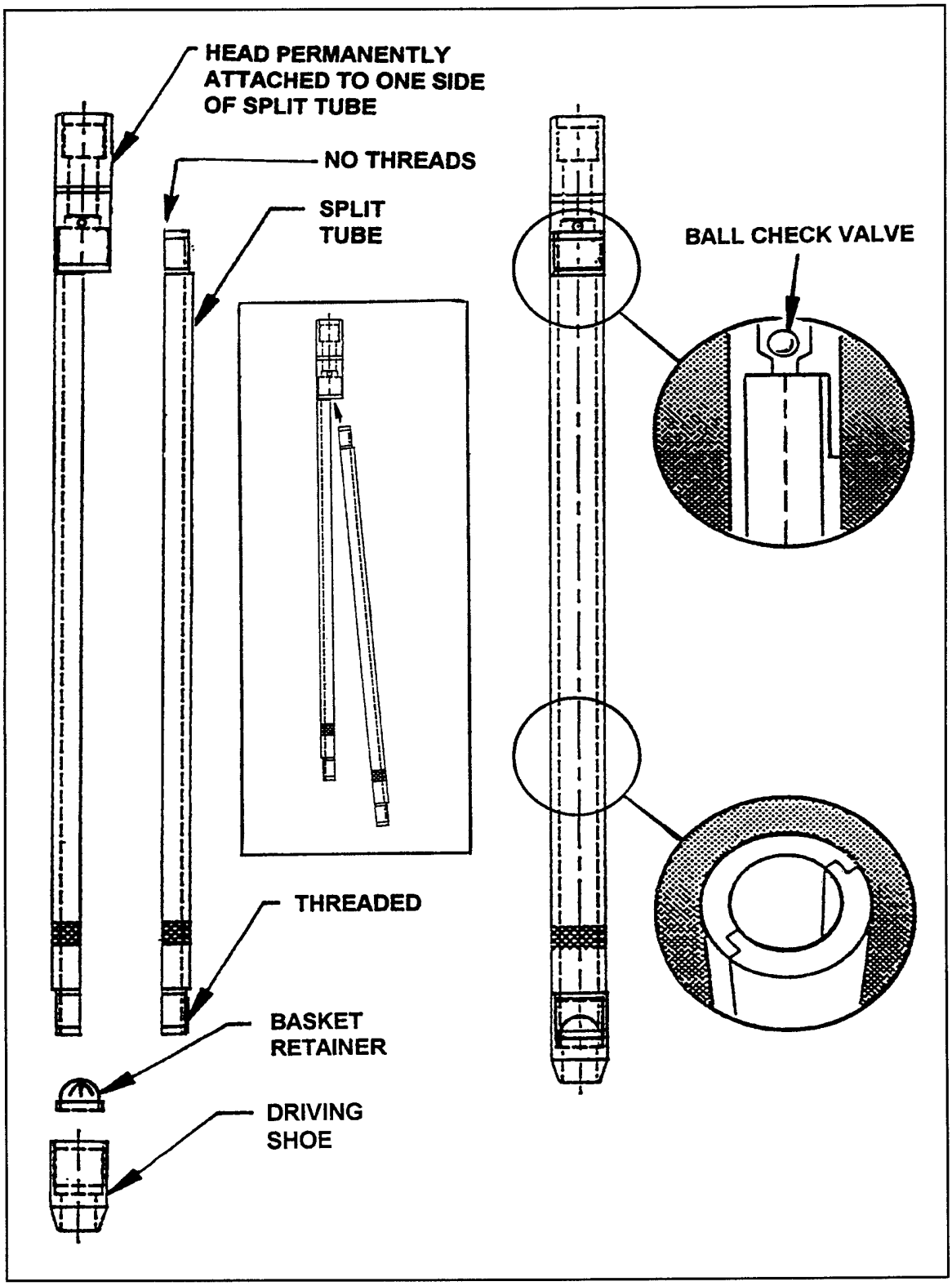


Figure C-17. Sanford quick-connect split-spoon sampler

- Complete all chain-of-custody documents and record information in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate sampling equipment after use and between sampling locations.

C.6.4.4 Ring-lined barrel sampler. Method Reference: ASTM D 3550.

C.6.4.4.1 Applicability. The ring-lined barrel sampler provides the ability to collect samples without losing volatiles or moisture. Soil is contained in the rings, and the sampler can be easily and quickly capped after it is removed. The relatively small size of the rings allows easy sample shipping and handling. However, the opportunity for describing the soil is diminished because most of the soil is concealed in the ring apparatus. Since rings are not always accepted by the laboratory, prior arrangements should be made with the laboratory.

C.6.4.4.2 Method summary and equipment. Ring-lined barrel samplers are typically 7.5 cm (3 in.) in diameter and are used to obtain representative subsurface soil samples with a split sampling barrel that has removable rings. The rings are typically constructed of plastic, stainless steel, or brass and fit inside the barrel assembly. Rings are commonly used within the California Modified sampler and are typically 7.5 cm (3 in.) long.

C.6.4.4.3 Sampling procedure. The sampling procedure is as follows:

- Place plastic sheeting on the ground around the sampling location to prevent cross- contamination.

- Assemble the sampler by placing eight 7.5-cm- (3-in.-) long rings in the 60-cm- (2-ft-) long sampler. Align both sides of the barrel and screw the drive shoe on the bottom and the heavier headpiece on top.

- Place the sampler in a perpendicular position on the material to be sampled.

- Drive the tube, using a sledge hammer or drill rig if available. Do not drive past the bottom of the headpiece because this will result in compression of the sample.

- Record the length of the tube that penetrated the material being sampled and the number of blows during each 15-cm (6-in.) increment.

- Withdraw the sampler and open it by unscrewing the drive shoe and head and the splitting barrel. Remove the sampling rings. Trim the soil at the end of the rings so that it is flush with the endings. For chemical samples, cap the end of the rings with a PTFE-lined plastic cap. For geotechnical samples, a plastic cap is suitable.
- Label the sample ring with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Place sealed sample rings on ice immediately.

- Complete all chain-of-custody documents and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate sampling equipment after use and between sampling locations.

C.6.4.5 Thin-walled (Shelby) tube sampler. Method Reference: ASTM D 1587.

C.6.4.5.1 Applicability. Thin-walled tube samplers allow collection of undisturbed samples in cohesive-type soils (i.e., clays). They are used primarily for collecting soil samples for certain geotechnical tests. Thin-walled tube samplers are not the ideal containers for transporting samples to the laboratory for chemical analysis due to their size. The opportunity for describing the soil is diminished because most of the soil is concealed in the tube.

C.6.4.5.2 Method summary and equipment. The thin-walled tube sampler is designed to take undisturbed samples in cohesive-type soils (Figure C-18). The thin-walled tube sampler is available in either brass, galvanized steel, plain steel, or stainless steel and is manufactured in either 76- or 91-cm (30- or 36-in.) lengths. These tubes normally have an outside diameter of 7.5 to 12.5 cm (3 to 5 in.); however, the 7.5-cm (3-in.) diameter is the most commonly used. Thin-walled tube samplers are usually used for sampling cohesive soils for geotechnical evaluation, rather than chemical analysis.

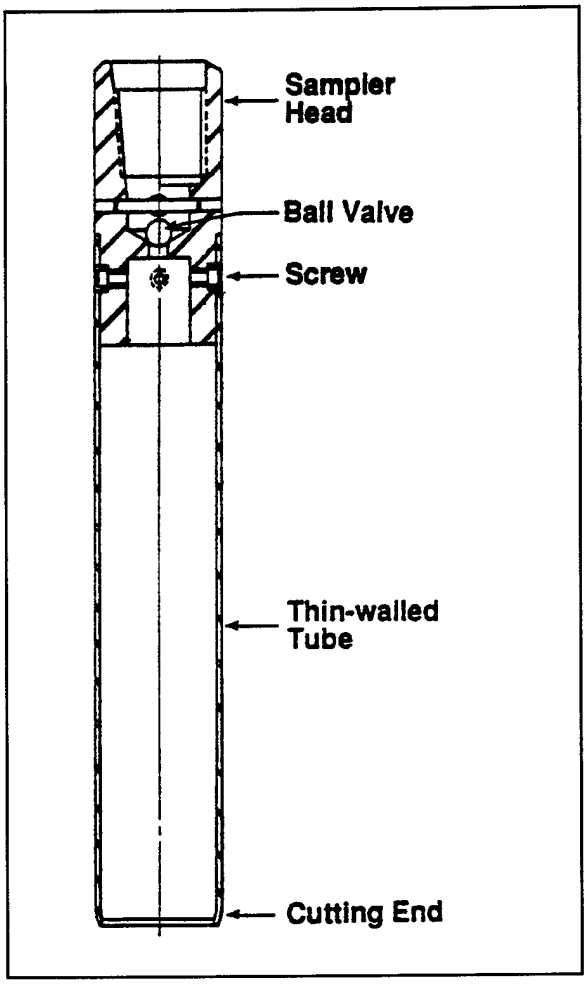


Figure C-18. Standard thin-walled (Shelby) tube sampler

C.6.4.5.3 Sampling procedure. The sampling procedure is as follows:

- Place plastic sheeting on the ground around the sampling location to prevent cross-contamination.
- Place the sampler in a perpendicular position on the material to be sampled.
- Push the tube into the soil by a continuous and rapid motion, without impact or twisting. In no instance should the tube be pushed further than the length provided for the soil sample.
- When the soil is so hard that a pushing motion will not penetrate the sample sufficiently for recovery, it may be necessary to collect a disturbed sample with the split-spoon sampler. Extremely dense and hard soils may result in damage to the thin-walled tube sampler.
- Before pulling out the tube, rotate the tube at least two revolutions to shear off the sample at the bottom. For geotechnical analysis, seal the ends of the tube with wax or rubber packers to preserve the moisture content. In such instances, the procedures and preparation for shipment

should be in accordance with ASTM D 1587. For chemical samples, seal the ends of the tube with PTFE-lined plastic caps.

- Label the sample tube with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Complete all chain-of-custody documents and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate sampling equipment after use and between sampling locations.

C.6.4.6 Continuous tube sampler. Method Reference: ASTM D 4700.

C.6.4.6.1 Applicability. The continuous tube sampler provides good samples for describing soil profiles because of the long length of the samples. Discrete samples for chemical analysis can be collected only within a 1.5-m (5-ft) increment. This sampler may not be effective in noncohesive soil types and requires the use of a drilling rig.

C.6.4.6.2 Method summary and equipment. The continuous tube sampler fits within a hollow-stem auger and is prevented from rotating as the auger is turned. The sampling tube can be split or solid barrel and can be used with or without liners of various metallic and nonmetallic materials. The sampler is illustrated in Figure C-19, and is typically 1.5 m (5 ft) long and 5 to 15 cm (2 to 6 in.) in diameter.

C.6.4.6.3 Sampling procedure. The sampling procedure is as follows:

- Place plastic sheeting on the ground around the sampling location to prevent cross- contamination.

- Lock the sampler in place inside the hollow-stem auger with its open end protruding a short distance beyond the end of the auger.

- Advance the auger while soil enters the nonrotating sampling tube.

- After advancing the length of the sampling tube, withdraw the sampler and remove the liner (if used) and cap. If a split-tube sampler is used, and chemical samples are desired, use a decontaminated stainless steel knife to divide the split-tube contents in half longitudinally.

- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.

- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing.

- Thoroughly mix remaining sample as outlined in Instructions E-2 or E-3 as appropriate and collect the sample into an appropriate sample bottle with a stainless steel laboratory spoon or equivalent.

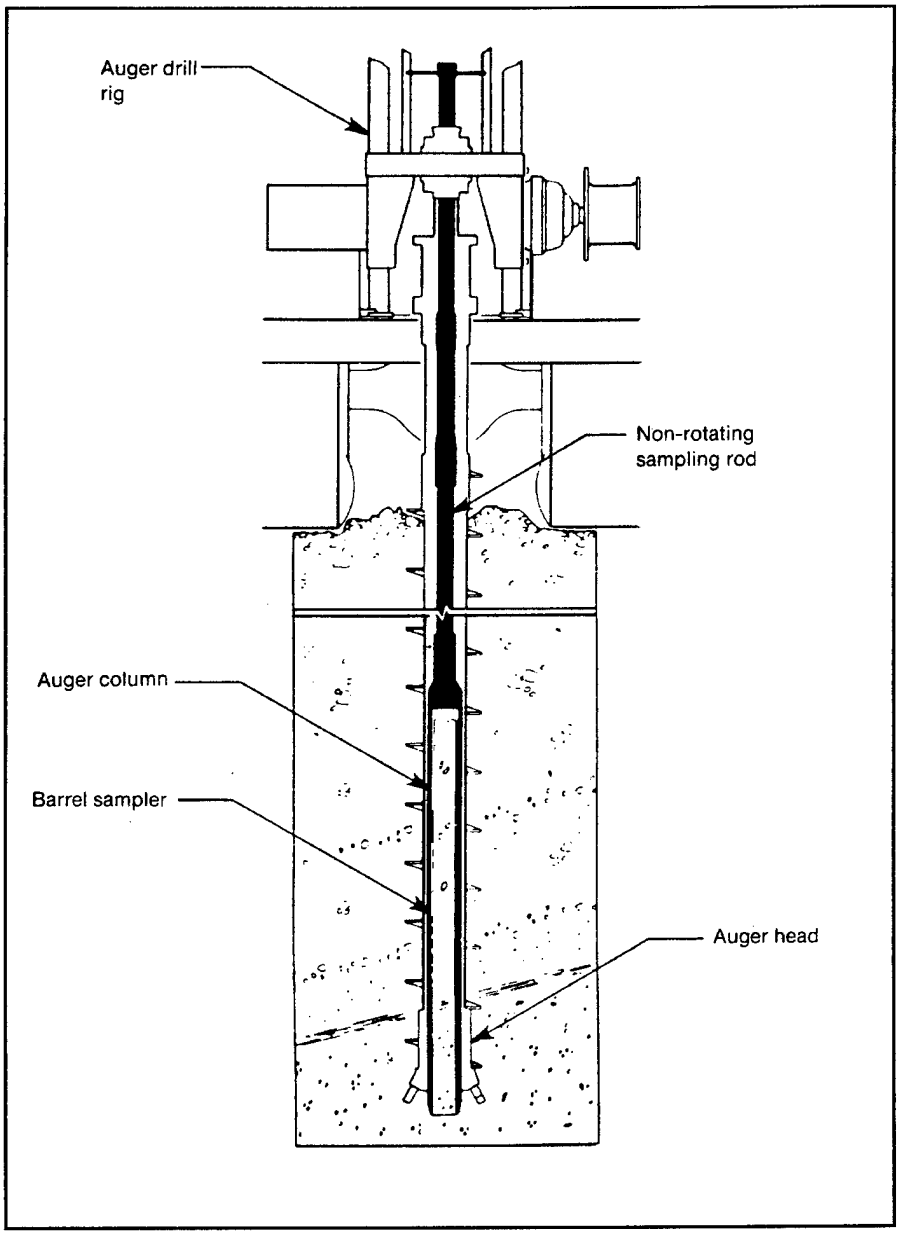


Figure C-19. Continuous tube sampler

- Secure the cap tightly.
- Place filled sample containers on ice immediately.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Complete all chain-of-custody documents and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate sampling equipment after use and between sampling locations.

C.6.4.7 Piston sampler. Method Reference: ASTM D 4700.

C.6.4.7.1 Applicability. Piston samplers are used to collect soft subsurface soils that cannot be collected using other techniques.

C.6.4.7.2 Method summary and equipment. The piston sampler (Figure C-20) consists of a sampling barrel with a piston that is retracted during sampling. Retraction of the piston creates a vacuum within the sample barrel that aids in retaining the sample in the barrel. Various piston type samplers are available, and each should be operated per the manufacturer's recommendations. EM 1110-1-1906 and EPA/625/R-93/00a discuss the use of a piston sampler.

C.6.4.7.3 Sampling procedure. The sampling procedure is as follows:

- Assemble decontaminated piston sampler and attach to rods that will lower the sampler down the borehole.

- Lower sampler to the desired depth. Advance the sampler into the soil while actuating the piston to create a vacuum within the sample barrel.

- Carefully remove the piston sampler from the bore hole.

- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.

- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing

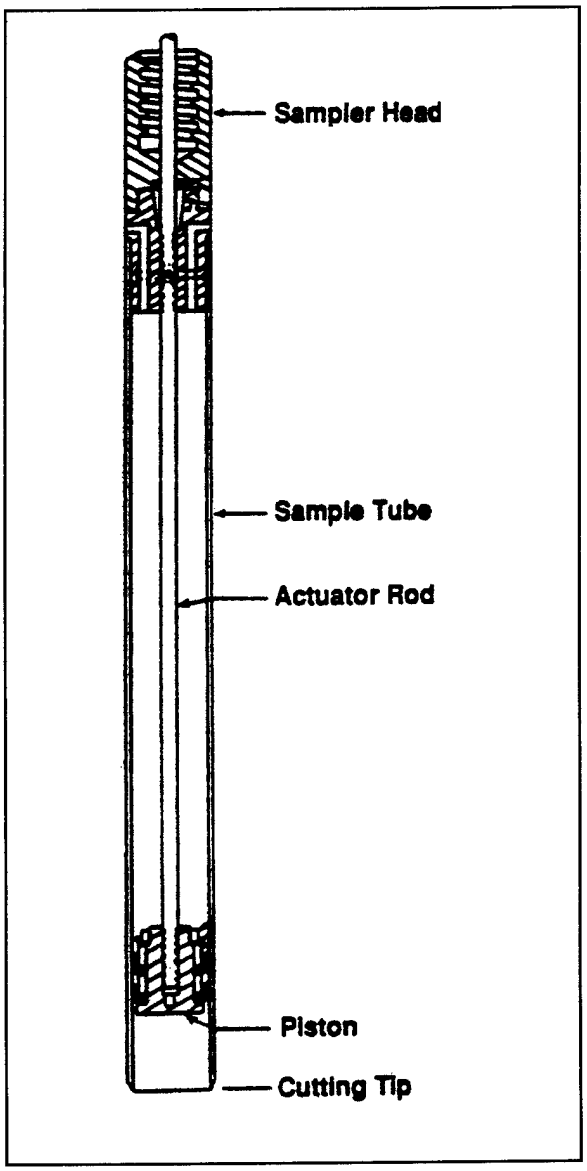


Figure C-20. Piston sampler (after ASTM D 4700, reprinted, with permission, from the Annual Book of ASTM Standards, copyright American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959)

of different locations is desired, transfer the sample to a stainless steel bowl for mixing. Refer to Instructions E-2 and E-3, Appendix E, respectively, for procedures on these techniques.

- Collect the sample into an appropriate sample bottle with a stainless steel laboratory spoon or equivalent.

- Label the sample bottle with the appropriate sample label. Complete the label carefully and clearly, addressing all the categories or parameters.

- Place the sample in an appropriate container and put the container on ice.

- Complete all chain-of-custody documents and field sheets and record in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Thoroughly decontaminate the sampler after each use.

C.6.4.8 Core barrel. Method Reference: ASTM D 2113.

C.6.4.8.1 Applicability. Core barrel or rotating core samplers advance by cutting away rock or soil material using a circular cutting bit as the shoe of the stationary inner core barrel advances into the rock or soil. Core barrel sampling is used primarily for collecting samples for rock profiling purposes. Rock samples are not typically submitted for chemical analysis.

C.6.4.8.2 Method summary and equipment. Core barrel drilling is used to obtain samples of rock or soils that are too hard to sample by soil sampling methods. Double-tube core barrels work the best. Core bits used for this type of sampling are typically impregnated with diamonds or carbide slugs that cut through the formation allowing a continuous sample to be collected.

C.6.4.8.3 Sampling procedure. The sampling procedure is as follows:

- Place the core barrel into position with the bit touching the ground or the surface to be cored.

- Continue core drilling until core blockage occurs or until the net length of the core barrel has been drilled.

- Remove the core barrel from the hole and disassemble it as necessary to remove the core.

- Refer to ASTM D 5079 "Preserving and Transporting Rock Core Samples" and EM 1110-1-1906 for sample handling procedures.

- Place the recovered core in a core box with the upper end of the core at the upper left corner of the core box. Cores should be placed in the core box as a book would read, from left to right and top to bottom, within the longitudinal separators. Space blocks or plugs should be securely placed at the beginning of each core run. The space blocks should be plainly marked with the depth of the core run. Spacers should also be securely placed in the proper positions in the core boxes and properly marked to show the location and actual extent of any voids and/or core losses during drilling.

- When a hole is completed, a space block marked "Bottom of Hole," or "BOH" should be securely placed after the last core run. Appropriately marked space blocks should also be inserted in the core boxes to fill the spaces formerly occupied by core that has been removed for testing.

- Core boxes should be marked on the outside to indicate the top and bottom, and the inside upper left corner of the box should be permanently marked with the letters UL to indicate the upper left corner.

- Soft or friable cores should be wrapped in plastic film or sealed in wax.

- When samples are collected with a core barrel and placed into a core box, the core samples should be photographed in the core box as soon as possible after the core samples are retreived and the box is labeled.

- The core box lid should be marked both inside and outside with the project name, hole number, location, surface elevation, box number, and depths for the beginning and end of core in the box. The ends of the core box should be marked with the project name, hole number and box number.

C.6.4.9 Direct Push Soil Sampling. Method Reference: ASTM D 6282.

C.6.4.9.1 Applicability. The direct push soil sampling method is widely used as a preliminary site characterization tool for the initial field activity of a site investigation. Direct push sampling is an economical and efficient method for obtaining discrete soil and water samples without the expense of drilling and its related waste cuttings disposal costs.

C.6.4.9.2 Method summary and equipment. The sampling method, known as the direct push method, involves sampling devices that are directly inserted into the soil to be sampled without drilling or borehole excavation. Direct push sampling consists of advancing a sampling device into the subsurface by applying static pressure, impacts, or vibration or any combination thereof to the aboveground portion of the sampler extensions until the sampler has been advanced its full length into the desired soil strata. No specific guidance or standards document the "direct push sampling method," but the guidance is a modification of standards from the Shelby tube, split spoon, piston, and penetrometer methods. The method is employed under various protocols by commercial entities and called by various proprietary names (i.e., Geoprobe). Direct push methods may be used to collect soil, and in some cases, the method may be combined with sampling devices capable of water and/or vapor sampling. The equipment generally used in direct push sampling is small and relatively compact allowing for better mobility around the site and access to confined areas. Direct push insertion methods include static push, impact, percussion, other vibratory driving, and combinations of these methods using direct push equipment adapted to drilling rigs, cone penetrometer units (the reference standard for which is ASTM D 5778-95, and specially designed percussion/direct push combination machines. Standard drilling rods used for rotary drilling are sometimes used when sampling is done at the base of drill holes. A direct push soil sampling system consists of a sample collection tool; hollow extension rods for advancement, retrieval, and transmission of energy to the sampler; and an energy source to force penetration by the sampler.

C.6.4.9.3 Sampling procedure. The sampling procedure is as follows:

- Assemble decontaminated direct push sampling device that will be pushed into the ground to collect data or samples.

- Advance the sampling device into subsurface soils by applying static pressure, impacts, or vibration or any combination thereof to the aboveground portion of the sampler extensions until the sampler has been advanced its full length into the desired soil strata.

- Sampling can be continuous for full-depth borehole logging or incremental for specific strata sampling. Samplers used can be protected for controlled specimen gathering or unprotected for general data collection.

- Recover the sampler from the borehole and remove the soil sample from the sampler.

- Begin sampling with the acquisition of any VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.

- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of a different location is desired, transfer the sample to a stainless steel bowl for mixing. Refer to Instructions E-2 and E-3, Appendix E, respectively.

- Transfer sample into an appropriate sample bottle using a stainless steel spoon or equivalent.

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

- Label the sample bottle. Complete the label completely and clearly, addressing all the categories and parameters.

- Place filled sample containers on ice immediately.

- Complete chain-of-custody documents and field sheets and record in the logbook (see Instruction F-1, "Documentation," Appendix F).

- Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate the equipment following each probe or sample.

C.6.4.10 Site Characterization and Analysis Penetrometer System (SCAPS).

C.6.4.10.1 Applicability. The SCAPS system provides the capability to conduct rapid site characterization and real-time analysis of contaminated soil and ground water in situ. SCAPS may be used to determine areas free of contamination, optimize the selection of monitoring well locations, and provide onsite three-dimensional visualization of soil stratigraphy and contaminant plumes. SCAPS site characterization may save from 25 to 30 percent of site characterization costs compared with those of conventional drill and sampling techniques. SCAPS sensors have the capability for determining soil classification/layering and detecting contaminants simultaneously. Geotechnical and contaminant sensing technologies currently available include soil classification; electrical resistivity; POL; explosives; VOCs; and gamma-emitting radionuclides. Regulatory certification has been granted for the SCAPS POL and is ongoing for VOCs, explosives, and metals sensors under the Environmental Security Technology Certification Program (ESTCP) and by the USEPA Consortium for Site Characterization Technologies, the California EPA Environmental Technology Certification Program, and reciprocity via the Interstate Technology Regulatory Cooperation

(ITRC) Workgroup. SCAPS technology has been transitioned to USACE, Department of Defense, Department of Energy, USEPA, and other government agency users and to the private sector through licensing and Cooperative Research and Development Agreements (CRADA).

C.6.4.10.2 Method summaries and equipment. The SCAPS platform consists of a 20-ton truck (Figure C-21) equipped with vertical hydraulic rams that are used to force a cone penetrometer into the ground at a speed of 2 cm/sec to depths of approximately 50 m in nominally consolidated fine-grained soils. During a vertical push, data are continuously collected and recorded with 2-cm depth resolution. The truck consists of two separate enclosed compartments (i.e., the data acquisition/processing room and the hydraulic ram/rod handling room). SCAPS multisensor penetrometer probes are equipped to measure tip and sleeve resistances simultaneously to determine soil stratigraphy, layer boundaries, and soil type as well as contaminant-specific sensor data to determine the presence of pollutants in each soil strata. SCAPS data acquisition consists of real-time data acquisition, electronic signal processing equipment, and a postprocessing computer for three-dimensional visualization of stratigraphy and contaminant plumes. The soil sampler used with the cone penetrometer consists of a lined steel cylinder with a retractable tip. The liner (typically a plastic type material) is placed in the sampler and the retractable tip is set at the bottom end of the sampler. The sampler is then advanced to the top of the interval where the soil sample is to be collected. The tip is remotely released and the sampler is pushed ahead into the interval to be sampled. Using this procedure, the soil sampler is pushed to the desired depth and the sample is collected without producing soil cuttings typically generated during soil boring activities. This type of soil sampler can be used with equipment other than cone penetrometers. A mobile laboratory truck may accompany the SCAPS to augment field analytical capabilities with the addition of an ion trap mass spectrometry and/or gas chromatography to analyze vapor samples collected by SCAPS samplers. In addition, the SCAPS can inject grout into the borehole through the probes as they are being withdrawn from the ground. This maintains isolation of contaminants in the subsurface.

C.6.4.10.3 Sampling procedures. The sampling procedures are as follows:

- Assemble decontaminated cone penetrometer device that will be pushed into the ground to collect data or samples.
- Push the data collection tip to the desired depth and record the data on the onboard computer. For the soil sampler, advance the sampler to the top of the interval to be sampled, release the tip, and advance the sampler to collect the soil sample.
- Following removal of the soil sample, backfill the hole by pumping grout through the tip as it is retracted, using the tremie method, or by pouring the grout into the hole from the ground surface.
- Remove the liner from the soil sampler and begin sampling with the acquisition of any VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4 for additional information on the collection, handling, and storage of solid VOA samples.
- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of a different location is desired, transfer the sample to a stainless steel bowl for mixing. Refer to Instructions E-2 and E-3, Appendix E, respectively, for procedures to follow for these techniques.
- Transfer sample into an appropriate sample bottle using a stainless steel spoon or equivalent.

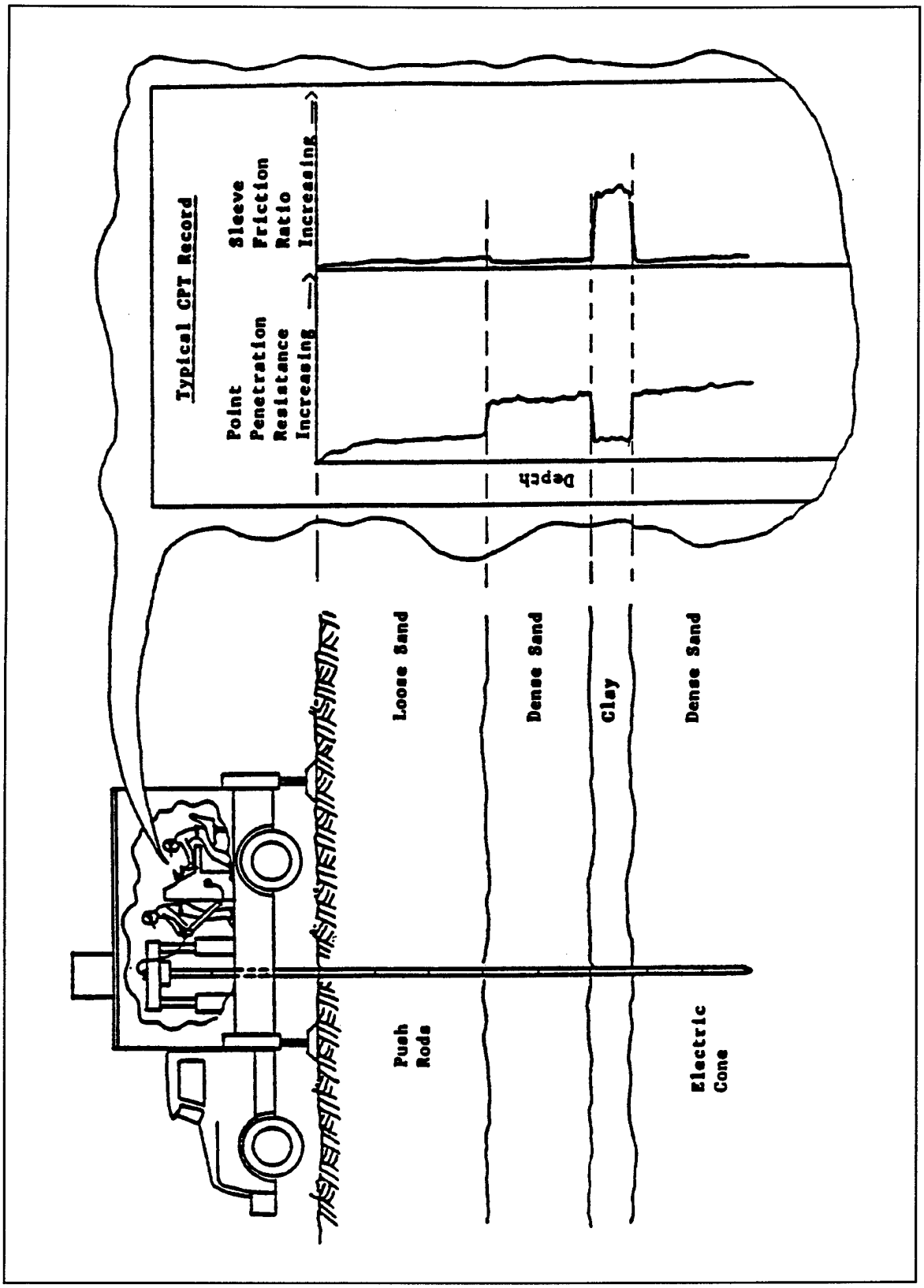


Figure C-21. Site characterization and analysis penetrometer system (SCAPS) truck schematic (CPT = cone penetrometer testing)

- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle. Complete the label completely and clearly, addressing all the categories and parameters.
- Place filled sample containers on ice immediately.
- Complete chain-of-custody documents and field sheets and record in the logbook (see Instruction F-1, "Documentation," Appendix F).
- Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate the equipment following each probe or sample.

C.6.4.10.4 Field analysis capabilities. The SCAPS has the following capabilities:

- Thermal Desorption VOC Sampler: detects and maps solvent and hydrocarbon contamination in both the vadose and saturated zones. Extracts vapor from in situ soil at penetrometer tip and retrieves vapor samples to surface for analysis.
- Hydrosparge VOC Sensing System: samples ground water by direct push tool, strips VOC from ground water by helium flow ("sparging"), and retrieves VOCs for analysis in ion trap mass spectrometer or other device.
- Explosives Sensor: incorporates electrochemical sensors inside the tip for analyzing unexploded ordnance in the contaminated strata; incorporates geophysical sensors for soil classification simultaneous with chemical analyses output at surface.
- Multiport Sampler (MPS): contains vertically stacked sampling modules independently operated from the surface for collecting multiple vapor samples in a single penetration without cross-contamination; alternative immediate retrieval of collected samples or retention for later retraction of probe.
- Laser Induced Fluorescence (LIF) Petroleum, Oil, and Lubricant (POL) Sensor: uses ultraviolet laser to induce fluorescence in POL contaminants in situ; returned fluorescent energy processed for spectral analysis of components in POL.
- Laser Induced Breakdown Spectroscopy (LIBS): a high-power focused laser creates a plasma (dissociates molecules and atoms in soil and energizes them) that emits electromagnetic radiation, which can then be analyzed by spectrometer for atomic content.
- X-Ray Fluorescence (XRF) Metals Sensor: uses x-ray excitation to fluoresce metal atoms independent of chemical states; characteristic energy emissions identify specific metals to well below 100 ppm concentrations.
- Enhanced Spectral Gamma Sensor: detects gamma radiation from radio nuclides for identification by gamma ray spectroscopy.

C.6.5 Decontamination procedures. All sampling equipment must be decontaminated prior to its use. Sampling equipment should be decontaminated as described in Instruction E-5 (Appendix E). The sampling equipment should be placed in plastic bags until immediately prior to use. Additional sampling devices may be needed onsite to ensure an adequate drying time.

C.6.6 Field control sample requirements. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They include blanks, replicates, and/or background samples. QA samples are replicates sent to a referee (QA) laboratory and analyzed to evaluate the contractor's laboratory performance. QC samples are blind replicates collected by the sampling team for analysis by the primary laboratory. A detailed discussion of field control samples is contained in Instruction G-2 (Appendix G).

C.6.7 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site set up, sample collection and handling as outlined above and in Instruction F-1, Appendix F.

C.7 Surficial Sampling

C.7.1 Scope and application. This instruction presents guidelines for collecting representative samples from various surfaces. Surficial sampling is used to assess the existence and/or extent of contamination on various surfaces rather than in a soil, water, or air matrix. For example, the contamination of the interior of a building may be assessed by collecting wipe samples of the process vessels and ventilation ducts. Surface samples are not typically analyzed for VOCs. Typical sample parameters include PCBs, dioxans/furans, pesticides, semivolatiles, metals, and explosives. Surface samples are typically divided into three media: nonporous surfaces, porous surfaces, and dust/soot. Nonporous surfaces can be sampled by wipe sampling; porous surfaces can be sampled by chipping or coring the surface; and dust/soot can be sampled by vacuum or sweep sampling. Instructions for these techniques are included in this section. If these methods are difficult to implement due to irregular surface shapes or other limitations, a rinsate sample can be collected.

C.7.2 Sampling strategies. The data from surficial sampling are typically required for risk assessments or compliance issues. Therefore, the sampling strategy is based either on a biased approach to locate and/or identify contamination or a systematic approach for decontamination verification.

C.7.2.1 Sampling locations. Sampling at hazardous waste sites is usually conducted in an attempt to discover contamination and to define its extent and variability. With such an objective, it is most logical to choose sample locations that will yield the most information about site conditions. Surficial sampling can be conducted by either biased or systematic sampling. Biased samples are those collected at locations that were chosen based on historical information, knowledge about the behavior of the contaminant(s), and/or knowledge about the effects of the physical system on the fate of the contaminant. Specific requirements for selecting sampling locations may be applicable for risk assessments or verification of cleanup levels. For example, sampling locations for verification of PCB cleanup levels are established in 40 CFR 761. Additional guidance for selecting sampling locations can be found in EPA/600/2-85/028, EPA/560/5-85/026, and EPA/560/5-86/017.

C.7.2.2 Types of samples. Surficial samples are discrete samples. Discrete (grab) samples are defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected immediately and at one particular point in the sample matrix. Representativeness of such samples is defined by the nature of the materials being sampled.

C.7.2.3 Suggested samplers. Each sampling technique presents various disadvantages and advantages for its application. For example, sample disturbance, sample volume, compatibility with planned chemical analysis, and chemical/physical reactivity between potential contaminants and sampling tool materials vary from technique to technique. Discussions of the advantages and disadvantages of each sampling technique are presented in the following sections.

C.7.2.4 Sample frequency. Determination of the number of samples needed to characterize a site also depends on the objectives and the site-specific conditions. For example, if the objective of the event is to determine whether site equipment is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, the site structures are known to be contaminated and delineation of the contamination is the objective, a greater number of samples may be needed. Confirmatory sampling for the effectiveness of the decontamination procedure may warrant specific sample frequency requirements. In many cases statistical considerations can be helpful in determining sampling strategy.

C.7.3 Sample preservation and handling. Many of the chemical constituents and physicochemical parameters that are to be measured or evaluated in investigation programs are not chemically stable; therefore, sample preservation is required. Chip, core, sweep, and vacuum samples should be handled in the

same fashion as sediment/soil samples. Wipe samples do not designate a specific preservation technique, but the use of amber glass may provide protection from light, and/or cooling may be employed as precautionary measures. Appropriate preservation techniques for various parameters are specified in Appendix B. In addition, sample containers that the sampler should use for each constituent or common set of parameters are specified in Appendix B. These preservation methods and sample containers are based on EPA/SW-846. Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2, "Packaging and Shipping Procedures," Appendix F.

C.7.3.1 Sample containers. Chip, core, and sweep samples should be placed in sample jars as designated for soil samples by the chemical parameters of interest. Wipe samples should be placed in amber jars. The cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Vacuum samples involve the acquisition of dust onto an air monitoring filter cassette, which includes the appropriate pore size (i.e., 0.8 micron) polycellulose acetate filter. Post sampling, end plugs are secured on the filter cassette for shipment to the laboratory.

C.7.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and to reduce sorption effects. Preservation methods are generally limited to refrigeration and protection from light. The sampler should refer to Appendix B or the specific preservation method in EPA/SW-846 for the appropriate preservation technique.

C.7.3.3 Special precautions for trace contaminant sampling. Contaminants can be detected in the parts per billion and/or parts per trillion range. Therefore, all equipment must be thoroughly inspected for cleanliness prior to use and extreme care be taken to prevent cross-contamination of these samples. The following general precautions should be taken when sampling:

- A clean pair of new, disposable gloves and shoe covers, if necessary, should be worn each time a different location is sampled. Gloves should be donned immediately prior to sampling. Remove and immediately dispose of shoe covers and gloves when sampling is complete.

- Avoid disturbing or tracking dust between locations by identifying and clearly marking all sampling locations upon arrival at the sampling site, and avoiding walking through or disturbing the marked areas prior to sampling.

- The appropriate template size should be identified within the project Sampling and Analysis Plan (SAP). Templates may be made of reusable aluminum or plastic or disposable cardboard or plastic. All templates should be thin (<3 mm (1/8 in.)), be able to lie flat, and must have accurately known inside dimensions. If reusable templates are employed, they must be decontaminated between sample (or subsample) locations.

- If appropriate, the solvent (including the required grade) in which the contaminants are most soluble should be identified.

- Sample containers should not be opened until needed to collect each sample. Sample containers for source samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting, preserving, tagging, etc.

- If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated

samples should never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants should be lined with new, clean, plastic bags. In general, sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.

- If possible, one member of the field team should take all the notes, fill out sample tags, field sheets, etc., while the other members collect all of the samples. The exact areas sampled should be recorded in the logbook.

- Adequate field control samples should be collected. For vacuum and wipe samples, a blank is mandatory to identify potential interferences from the filter/gauze, solvent (wipe only), or sample containers.

C.7.4 Sampling methods. The following are sampling instructions for the most common techniques for collecting surface samples.

C.7.4.1 Surface wipe sample.

C.7.4.1.1 Applicability. This method of monitoring surficial contamination is intended for nonvolatile species (e.g., PCBs) on nonporous surfaces (e.g., metal, glass). Sample points should be carefully chosen and should be based on site history, manufacturing processes, personnel practices, obvious contamination, and available surface area.

C.7.4.1.2 Method summary and equipment. Surface wipe sampling methods vary and are dependent on the data objectives.

C.7.4.1.3 Sampling procedure. A generalized procedure is presented here for reference.

- Place a clean, appropriately sized square template cutout over the area to be sampled (Toxic Substances Control Act (TSCA) requires 100 cm^2). Secure the template to avoid any shifting during the sampling activity. Do not touch or walk on marked areas. Areas should be documented by a drawing of the floor plan, if applicable, in the field logbook. Unique sample number identifiers should be included within the drawing.

- Remove a gauze pad from the box of gauze using decontaminated tongs (filter paper may also be used). Be sure to use a new pair of surgical gloves.

- Soak the gauze or filter pad in appropriate solvent.

- Using a decontaminated pair of tongs, wipe the area framed by the template cutout with the moistened gauze in one direction.

- Without allowing the gauze to contact any other surface, fold the gauze with the exposed side in, and then fold it again to form a 90-degree angle in the center of the gauze.

- Place the gauze in an amber laboratory sample container, angle first, and replace the container cap.

- Secure the cap tightly.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label properly and clearly, addressing all categories or parameters.

- Place a filled sample container on ice immediately, if desired.

- Complete all chain-of-custody documents and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare the samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Mark the area of the cutout using a paint stick, if possible.

- Record the location data, station number, sample time, date, and names of the sampling crew in the field logbook or log sheet for each wipe sample. In addition, document the sampling locations by including a drawing or dimensioned sketch in the field logbook or log sheet if the sampled area cannot be marked by a paint stick or if locating the area from the field notes would be difficult.

- Dispose of generated waste material properly.

C.7.4.2 Chip/core sample.

C.7.4.2.1 Applicability. This method of monitoring surficial contamination is intended for nonvolatile analytes (e.g., PCBs) on porous surfaces (e.g., cement, brick, wood). Suggested sampling points include floors near process vessels, storage tanks, loading docks, etc.

C.7.4.2.2 Method summary and equipment. Samples from porous surfaces can be obtained by breaking up a designated surface with a chisel, brushing up the chipped pieces, and transferring the sample into a bottle. A core sample can also be collected using appropriate power tools. However, most confirmatory sampling requires that only the upper 6 mm (1/4 in.) of the media be sampled. Core samples may require further sample handling (i.e., sawing) to isolate this portion of the sample for analysis, or may risk diluting contaminants that may be present only in the upper 6 mm (1/4 in.) if the sample is analyzed as a whole, and are therefore discouraged.

C.7.4.2.3 Sampling procedure. Once the sample location has been determined, measured, and marked off, sample collection can begin as follows:

- Place a clean, appropriately sized template cutout over the area to be sampled. The template may be square, U-, or L-shaped, as appropriate to the area under assessment. Secure the template to avoid any shifting during the sampling activity. Do not touch or walk on marked areas. If applicable, areas should be documented by a drawing of the floor plan in the field logbook. Unique sample number identifiers should be included within the drawing.

- Use a decontaminated chisel and hammer to break up the surface to be sampled (TSCA requires 100 cm^2). Avoid scattering pieces. Chip the area to less than 6 mm (1/4 in.) in depth.

- Record the depth at which the chips were taken.

- Collect the chipped pieces using new clean gloves and a pair of decontaminated tongs.

- Transfer the sample directly into the sample bottle.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place the filled sample container on ice immediately.
- Complete all chain-of-custody documents and record in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

C.7.4.3 Vacuum dust sample.

C.7.4.3.1 Applicability. This sampling method is used to monitor surficial contamination for carpeted or noncarpeted areas. Sampling is typically intended to assess residential or industrial dust contamination associated with metals species (e.g., lead). Sample points should be carefully chosen and should be based on traffic areas, dust migration pathways, potential exposure of most susceptible personnel (e.g., the bedroom of youngest/oldest resident), obvious contamination, and available surface area. Undisturbed areas such as an attic may be used to assess historic dust concentrations.

C.7.4.3.2 Method summary and equipment. Surface dust samples are vacuumed onto a filter with the use of a calibrated air monitoring pump. Samples may be a composite of subsampled areas or individual samples taken from each location, depending on the project data objectives. All sampling locations should be clearly defined within the Field Sampling Plan portion of the SAP.

C.7.4.3.3 Sampling procedure. A generalized procedure is presented here for reference.

- Place a clean, appropriately sized template cutout over the areas to be sampled (e.g., 0.09 to 0.03 m^2 (1 to 3 ft^2)). The template may be square, U-, or L-shaped, as appropriate to the area under assessment. Secure the template to avoid any shifting during the sampling activity. Do not touch or walk on marked areas. Areas should be documented by a drawing of the floor plan in the field logbook. For composite samples, areas should be numbered to depict the sequence of sample collection. For individual samples, unique sample location identifiers should be included within the drawing.

  (1) For carpeted areas, suggest placing templates at any high-traffic area in the room (e.g., immediately inside doorways into room).

  (2) For noncarpeted (hard-surface) areas, suggest placing templates in areas where dust is likely to migrate (e.g., immediately adjacent to wall or at the edges of the room).

- Connect the inlet port of a portable, battery-operated air monitoring pump (capable of maintaining a flow rate of 3.0 L/min through a filter cassette) to tubing sized to fit tightly on the outlet side of the air monitoring filter cassette. Pump flow must be calibrated to the project-specified flow rate per manufacturer's instructions using a soap bubble airflow meter or calibrated rotameter.

- Connect the collection nozzle (i.e., stainless steel or carbon-impregnated plastic machined/molded to form a thin rectangular opening of 1.3 mm by 1.2 mm (1/2 in. by 3/64 in.) for the sampling end and the other machined/molded to accept tubing) to tubing sized to fit tightly on the inlet side of the filter cassette.

- Hold the vacuum in the sampling position above the template, turn on the air monitoring pump, and ensure the flow rate is at 3.0 ± 0.2 L/min (or as specified in project SAP).

- Execute the vacuuming technique as follows:

  (1) First pass: Hold the collection attachment at a 45-degree angle to the surface and move from one side of the template to the opposite side. The rate of movement should be approximately 1.5 to 2 seconds for each stroke. Continue in the same direction until the entire area has been vacuumed.

  (2) Second pass: Repeat the above procedure in a direction 90 degrees from the first pass.

  (3) Third pass: Repeat this procedure in the same direction as the first pass.

- Remove the filter (dust) cassette from the tubing attachments and secure with end caps.

- Label the filter (dust) cassette with pertinent sample information as defined in Instruction F-1, Appendix F.

- Complete all chain-of-custody documents and record information in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare the samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Record all measurements onto a drawing of the floor plan, including the location data, station number, sample time, date, and names of the sampling crew, in the field logbook or log sheet for each vacuum sample.

- Dispose of generated waste material properly.

C.7.4.4 Sweep sample.

C.7.4.4.1 Applicability. This method of monitoring surficial contamination is intended for nonvolatile analytes (e.g., PCBs) in the residue found in porous (e.g., asphalt) or nonporous (e.g., metal) surfaces. Sweep sampling allows collection of dust/residue that may help in the assessment of contaminant determination and delineations. Sample points should be carefully chosen and should be based on traffic areas, dust migration pathways, potential exposure personnel, obvious contamination, and available surface area. Undisturbed areas such as an attic may be used to assess historic dust concentrations.

C.7.4.4.2 Method summary and equipment. Dust and residue samples can be collected with a bristle brush and dustpan in places where solvents cannot be used or when large amounts of dust/residue make wipe samples impractical.

C.7.4.4.3 Sampling procedure. Once the sample location has been determined, measured, and marked off, sample collection can begin as follows:

- Put on clean, chemical-resistant gloves (separate pair for each location).
- Place a clean, appropriately sized template cutout over the area to be sampled. Templates may be square, U-, or L-shaped, as appropriate to area under assessment. Secure the template to avoid any shifting during the sampling activity. Do not touch or walk on marked areas. Areas should be documented by a drawing of the floor plan in the field logbook. For composite samples, areas should be numbered to depict the sequence of sample collection. For individual samples, unique sample location identifiers should be included within the drawing.
- Sweep all residues from the area to be sampled into the dustpan.
- Transfer the sample directly into the sample bottle.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Place a filled sample container on ice immediately.
- Complete all chain-of-custody documents and record in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

C.7.5 Decontamination procedures. All nondisposable templates and sampling equipment must be decontaminated prior to its use. Field equipment should be cleaned as described in Instruction E-6, Appendix E. The sampling equipment should be placed in a plastic bag until immediately prior to use. Additional sampling devices may be needed onsite to ensure an adequate drying time.

C.7.6 Field control samples requirements. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They include blanks, replicates, and/or background samples. A detailed discussion of field control samples may be found in Instruction G-2, Appendix G, and is summarized in the following sections as they are applied to surficial sampling.

C.7.6.1 Duplicate/split samples. True duplicate/split samples cannot be collected in a surficial or dust/soot sampling program. The sampling medium (i.e., filter or gauze) used to collect the sample cannot be divided to obtain a duplicate/split sample because the contaminants will not be spread evenly on the sample medium. The same surface area cannot be sampled a second time to obtain a duplicate/split sample because the first sample will remove the contaminants from the area under assessment. Collecting a sample from an area adjacent to the first sampling area is a viable alternative for collecting a duplicate/split sample. However, the sample is not a true duplicate/split sample and the contaminant concentrations in the samples from the adjacent area may not be the same.

C.7.6.2 Blank samples. Vacuum and wipe blanks are samples collected in the field to determine if any interference has been caused by the sample collection materials (i.e., filters/gauze, solvent (wipe only), or sampling equipment). These blanks are obtained by preparing the sample medium for sampling, placing the solvent on the gauze if applicable, and placing the sampling medium in the sample containers, or securing

the sample for shipment as directed within the instruction. The sample medium does not contact any sampling surface.

C.7.6.3 Background samples. Background samples are recommended to be taken in conjunction with chip/core samples. Background samples should be taken using the same procedures used to obtain the field samples. The samples should be obtained from an uncontaminated area of the same matrix used to collect the field samples. The rationale for collecting background samples is to determine if there are any interferences inherent in the porous matrix.

C.7.6.4 Rinsate blank samples. Rinsate samples consist of reagent water collected from a final rinse of surfaces after decontamination procedures have been performed. The purpose of the rinsate samples is to determine the thoroughness of the decontamination procedures performed.

C.7.7 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site setup, sample collection and handling as outlined in the preceding sections and in Instruction F-1, Appendix F.

C.8 Air and Soil Gas Sampling

C.8.1 Air sampling. This section addresses the aspects of gas phase sampling and analysis for HTRW projects. This type of sampling can involve four categories of sample collection and measurements: soil gas in situ or extractive; surface area emission flux; point source emissions or remediation process streams, including in-place control and containment systems; and ambient air at the site perimeter or at off-site receptor locations. The media sampled for soil gas or surface area emissions are typically gaseous phase samples. Point source, process streams, and ambient air sampling can involve either gaseous phase samples or particulate matter.

C.8.2 Soil gas sampling.

C.8.2.1 Scope and application. Soil gas monitoring of the vadose zone is used to measure characteristics of the soil atmosphere as an indirect indicator of contamination in or below the sampling horizon. The information may be used for health and safety purposes, to detect and monitor migration of volatile contamination of soils within the vadose zone, to support the design of a soil vapor extraction system, to assess the extent of ground water volatile organics contamination, to verify underground storage tanks integrity and monitor for any subsurface discharges, and to aid in locating ground water monitoring wells, extraction wells, recovery trenches, etc. It is also used for general reconnaissance, and as a screening technique when large areas are being evaluated that have little or no information available on past waste disposal practices. The primary limitation to this type of sampling is that it is not a stand-alone technique, and additional sampling of environmental media is necessary to determine the source and extent of contamination. Soil gas samples may be analyzed onsite, or samples containerized and shipped to a laboratory for offsite chemical analyses. The overall effectiveness of a soil gas sampling method to depict subsurface conditions accurately is dependent on several factors, including soil permeability; infiltration moisture; barometric pressure changes; volatility of the contaminant; the presence of any underground obstacles or conduits, pavements, or other features affecting pathways; the presence of interfering chemical compounds, etc. Soil gas sampling technology is most effective in mapping low-molecular-weight, halogenated, or aromatic hydrocarbons that possess high vapor pressures and low aqueous solubilities (e.g., benzene, toluene, trichloroethylene, trichloroethane, vinyl chloride, etc.)

C.8.2.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified during the HTRW technical planning process. Sampling strategies will vary based on the intended purpose of the soil gas survey; and they are significantly influenced by such factors as site physical constraints, soil types and stratigraphy, background information available, and type of contamination, to name a few. When soil gas sampling is used to optimize monitoring wells or soil boring locations or monitor the integrity of underground storage tanks, a sampling strategy based on a judgmental or biased approach is recommended. However, when soil gas sampling is used for general site reconnaissance, the use of a statistical sampling method (i.e., a systematic grid approach) is typically used. Many projects use a combination of these sampling strategies. For instance, an initial sampling may be done by a predetermined regularly spaced grid pattern to identify presence of contamination, followed by a judgmental or close, irregularly spaced grid pattern approach to refine the delineation of the contamination detected. Grid spacing shape and size should be based on the project objectives and allowable uncertainty in the decision-making process, but be flexible enough to allow modifications in the field to account for site characteristics, or generated results. The technical planning process that results in the development of project soil gas sampling strategy is critical because of the difficulty in acquiring representative soil gas samples.

C.8.2.3 Sampling locations. Sampling at hazardous waste sites is usually conducted in an attempt to discover contamination and to define its extent and variability. With such an objective, it is most logical to choose sample locations that will yield the most information about site conditions. As noted in the

preceding section, a soil gas sampling survey can be conducted by following random, systematic, or biased sampling strategies. The same information used to select the sampling strategy is used to choose the soil gas sampling locations. Sample locations must account for a variety of physical properties of the soil, including grain size, cohesiveness, organic matter, moisture content, geographic fractures, and overall soil permeability. In addition, the properties of the chemical contaminants must also be considered, including volatility, solubility or immiscibility in water, and degradation potential. Appropriate sampling depths are determined by assessing these factors and the depth to ground water, the source, soil stratigraphy, and features that enhance or impede the movement of the soil gas. Multiple depth sampling is also an option, when complex geologic settings are encountered.

C.8.2.4 Sampling techniques and sample types. Soil gas samples may be generated in conjunction with an active or passive sampling system. The samples themselves may be a whole air sample or a sorbent sample composed of an absorbent/adsorbent medium that collects contaminants during a specified time frame.

C.8.2.4.1 Active soil gas sampling system. An active soil gas sampling system involves the forced movement of bulk soil gas from the vadose zone to a collection device through a probe or similar apparatus by the influence of a vacuum pump. The air within the system may be monitored real-time through a sample port with an appropriate detector (e.g., photoionization detector or flame ionization detector) or coupled directly to an analytical system for direct monitoring readout documentation. Active soil gas systems may also be sampled with a gas-tight syringe for onsite gas chromatograph analysis, or whole air samples collected in tedlar bags, glass bulbs, summa canisters, or other containment devices for subsequent onsite or offsite analyses. Refer to Figure C-22 for an illustration of a summa canister for the collection of whole air samples. The active soil gas sampling system may also be configured to collect a sorbent sample. Refer to Figure C-23 for a schematic of a sorbent media cartridge for the collection of sorbent samples. In this way, the probe is connected to a collection device designed to extract and trap chemical contaminants from the airstream by adsorption. This technique is especially useful when very low-level contamination is present that is below the instrument sensitivity capabilities for whole air samples, and preconcentration of the contaminants onto the adsorbent medium is necessary. Some sorbent media can also be used to segregate interfering compounds (e.g., reactive or oxygenated organic compounds), for the medium can be treated or designed so the inferences will not be adsorbed or easily desorbed from the collection medium. Sorbent samples are also beneficial when the chemical contaminants are more amenable or more efficiently removed and detected by extraction techniques. The collection medium is typically activated charcoal, silica gel, activated alumina, various porous polymers, or molecular sieve adsorbents that have been prepared by thermal (or solvent) desorption treatment. Limitations in the use of sorbent samples include interactions between the

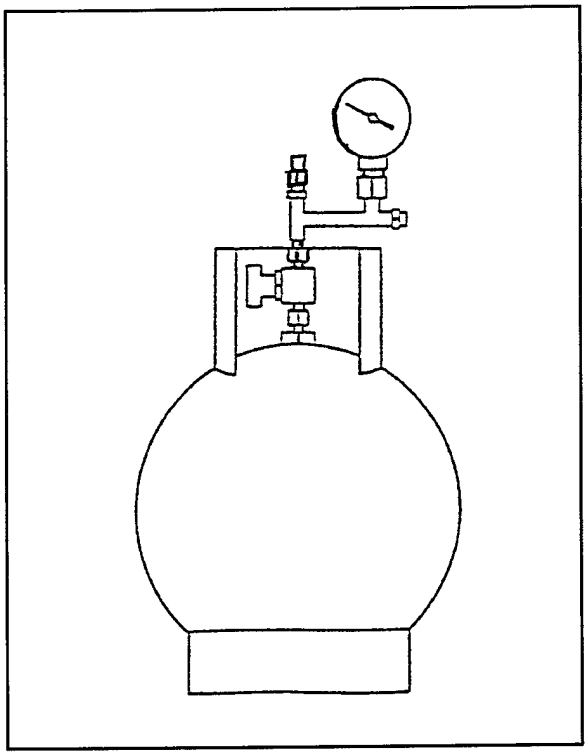


Figure C-22. Rigid whole air collection canister (summa canister)

contaminants and the sorption medium, the release of artifacts during the desorption process, and high humidity, which can significantly impact the adsorption efficiency of the sorption medium. Advantages in using an active soil gas sampling system include the quick turnaround for data and the ability to tailor the survey as it progresses. Limitations of an active soil gas system include its cost compared to that of passive techniques and its susceptibility to meteorological effects, i.e., false negative results when rainfall or snowmelt occurs. This is due to infiltration water driving the contaminant vapors ahead of the infiltrating front and drawing clean atmospheric air into the zone to be sampled. Driven probes also tend to degrade the natural soil permeability around the body of the probe due to soil compaction during probe insertion. This may severely impede the active soil gas flow in moist, heavy clay soils.

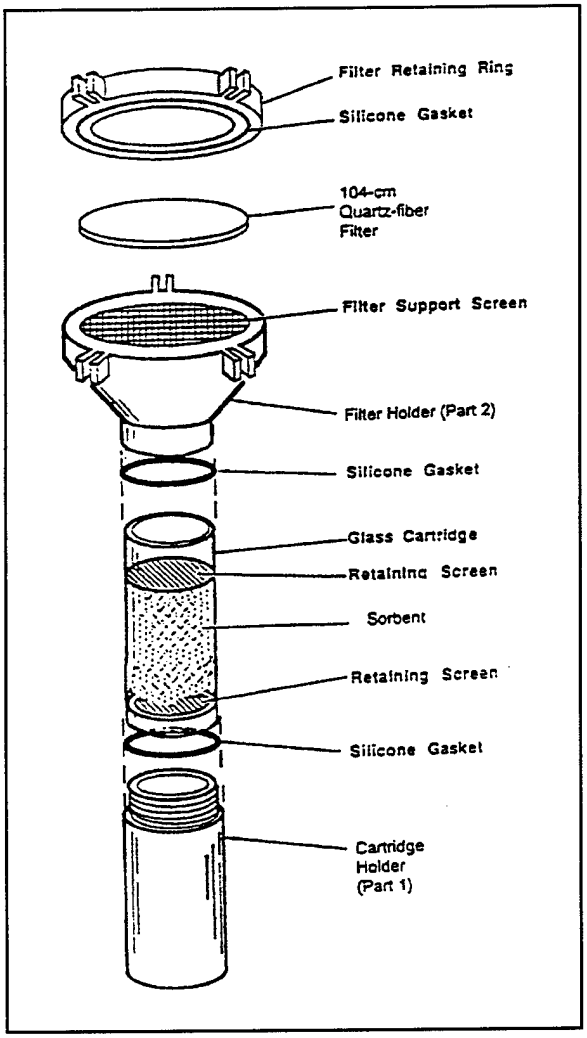


Figure C-23. Sorbent media (resin) cartridge

C.8.2.4.2 Passive soil gas sampling system. A passive soil gas sampling system entails placing a collection device in the subsurface or on the surface of the ground and allowing the atmosphere in the device to come to compositional equilibrium with the soil atmosphere. A passive soil gas sampling system may be used to collect whole air samples. This approach may be used to monitor contaminant emissions for health and safety purposes (i.e., methane emissions from a landfill) and is discussed in Section C.8.3. More commonly, the passive soil gas sampling techniques are used in conjunction with a sorbent medium. Several varieties of these collection devices exist, with slightly different configurations. However, the basis for sampling is the same, in that it is the passive movement of contaminants in soil to a sorbent medium contained in a collection device over a specified period of time. Several types of sorption media are available, which, due to their adsorption properties, will reduce the contaminant concentration directly adjacent to the device. This induced concentration sink helps maintain continuous migration of contaminants from the vadose zone toward the collection device by diffusion and mass transfer. The rate of contaminant migration is limited by many of the same factors affecting the active soil gas sampling techniques. These include soil physical characteristics and the physical and chemical properties of the contaminants. In addition, caution must be exercised during installation or backfilling to avoid cross-contamination of the sorbent media. Typical residence times for the collection devices range from several days to weeks to complete. The passive-sorbent sample system is not recommended when the ground is frozen or when saturated soil conditions exist. Advantages to the use of a passive soil gas sampling system include its nominal cost, its lower susceptibility to meteorological changes, and the addition of less volatile contaminants that may be analyzed. Limitations include the following: time frames for data retrieval are

long, which necessitates the need for more samples for thorough coverage, and it is not appropriate for use with soil vapor extraction system design.

C.8.2.5 Sample preservation and handling. Methods for preservation of whole air and sorbent samples are relatively limited and are generally intended to retard thermal and photodegradation. Preservation methods for whole and sorbent samples are typically avoidance of heat or light. This may be as simple as placing samples potentially exposed to light (i.e., tedlar bags) in dark plastic garbage sacks, and keeping out of direct sunlight. Sorbent samples preservation will vary based on the sorbent medium used. Therefore, follow any procedures identified by the vendor or manufacturer.

C.8.2.6 Collection of adequate field control samples. Field control samples are collected by the sampling team to determine whether the data are of suitable quality. They may include blanks, replicates, and/or background samples (atmospheric blanks). The application, type, and frequency of field control sample acquisition should be based on the purpose of the soil gas survey and the associated data quality requirements. Field control samples taken in conjunction with soil gas sampling techniques are typically limited to a variety of field blanks to assess potential cross-contamination sources and contribution. Field blanks used include trip (travel) blanks, sample container blanks, and sample probe blanks. Field replicates may be taken. However, due to the difficulty in acquiring representative replicate air or adsorbent samples, taking field replicates is not commonly practiced. A detailed discussion of field control samples is contained in Instruction G-2, Appendix G.

C.8.2.7 Sampling methods. Due to a variety of configurations for passive soil gas sampling methods, defer to the individual manufacturer's installation and instruction manual. The following encompasses general procedures for an active soil gas sampling system. Additional procedures may be necessary based on the manufacturer's specifications. Method Reference: ASTM D 5314.

C.8.2.7.1 Applicability. Active soil gas sampling systems are widely used as a preliminary site characterization tool for the initial field activity of a site investigation. Active and passive soil gas sampling systems are an economical and efficient method for obtaining information on potential subsurface contamination without the expense of drilling and its related waste cuttings disposal costs.

C.8.2.7.2 Method summary and equipment. The active soil gas sampling system involves sampling devices that are directly inserted into the soil to be sampled without drilling/excavation, or into the soil ahead of the auger or drill bit if performing deep sampling or vertical profiling. The active soil gas sampling consists of advancing a sampling device into the subsurface by applying static pressure, impacts, or vibration or any combination thereof to the aboveground portion of the sampler extensions until the sampler has been advanced its full length into the desired soil depth or strata. The equipment generally used to drive the sample probe is small and relatively compact allowing for mobility around the site and access to confined areas. The probe is then retracted slightly to remove the expendable drive point or open a sample port and create a void where soil gas may enter the sample probe. A vacuum is drawn on the system with a pump to induce the flow of soil gas through the sample probe to the surface where whole air or sorbent samples may be acquired.

C.8.2.7.3 Active soil gas sampling procedure. The sampling procedure is as follows:

- Assemble decontaminated active soil gas sampling probe device, consisting of a drive point, point holder, a hollow extension pipe, or other equipment as specified by a manufacturer. Push the apparatus into the ground to collect samples (Figure C-24).

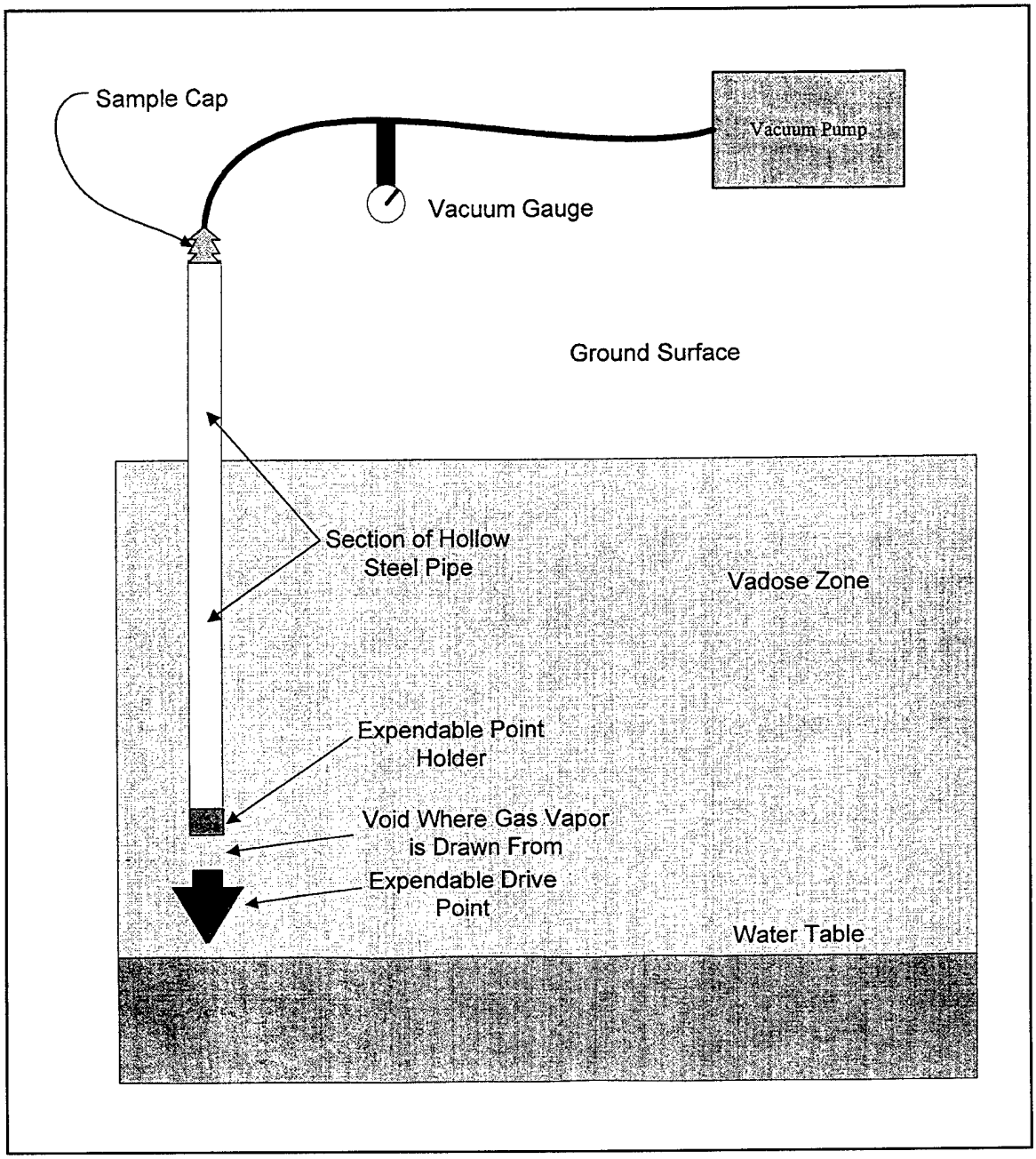


Figure C-24. Active soil gas apparatus

- Advance the sample probe by applying static pressure, impacts, or vibration or any combination thereof to the aboveground portion of the sampler extensions until the sampler has been advanced its full length into the desired soil depth or strata (typically 1.5 to 3 m (5 to 10 ft)).

- Sample probes may be fitted with detachable drive tips, which become removed from the steel pipe when the probe is retracted.

- Attach the flexible tubing with appropriate sampling port or sorbent media cartridge, followed with additional flexible tubing to a vacuum pump.

- Extract gas through the probe via the vacuum pump. Five probe volumes should be extracted prior to sampling. A qualitative check for leaks or short circuits is performed by monitoring the vacuum and flow rates. A drop in vacuum or too low a vacuum may indicate system problems. Quantitative leak checks may also be used to verify that the system performance is acceptable at the highest vacuum required by the project. Project-specific acceptance criteria should be established when necessary.

- Begin sampling with appropriate sample containers through a sampling port set between the probe and the pump. These may include real-time monitors (photoionization detector or flame ionization detector), gas-tight syringes, tedlar bags, summa canisters, etc., for a whole air sample. Or preset the appropriate sorbent media and cartridge at the same location between the probe and the vacuum pump.

- Close off appropriate valves for sample containers. Samples should be protected from heat and light.

- Label the sample containers. Complete the label completely and clearly, addressing all the categories and parameters.

- Complete chain-of-custody documents and field sheets and record in the logbook (see Instruction F-1, "Documentation," Appendix F).

- Samples not analyzed immediately should be prepared for shipment to the laboratory (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate the equipment following each probe or sample.

C.8.2.8 Decontamination procedures. All sampling equipment must be disposable or decontaminated prior to its use. Sampling equipment should be decontaminated as described in Instruction E-6 (Appendix E).

C.8.2.9 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site setup and sample collection, preparation, and handling, and all field analytical reportables (i.e., calibration data, sample and QC results, chromatograms, etc.) as outlined previously and in Instruction F-1, Appendix F.

C.8.3 Surface emission flux measurements

C.8.3.1 Scope and application. Surface emission flux rate sampling for gaseous compounds involves the isolation of either an "as-is" surface area or a prepared "scraped" area to collect and quantify the release of contaminants of concern. A flux chamber is used to cap the surface area being evaluated while the chamber is continuously swept by the injection of an inert gas or air. The resulting gaseous mixture is then sampled and analyzed. The rate of contaminant emission from the source area is calculated based on the flow rate of the incoming inert gas and the concentrations of contaminants found in the mixed gas sample. This type of sample collection and measurement is often selected when indoor or localized ambient air contamination is of concern due to contaminant migration from known or suspected HTRW sites. Emission

flux data are collected to provide more accurate site-specific contaminant emission data than may be obtained from predictive models. The calculated emission data can be used for contaminant fate and transport calculations, risk assessment, or determination of the rate of release to the atmosphere. In effect, this measurement may improve the accuracy of the "migration" rate from a source to the gaseous phases. The results of such measurements are expressed in units of contaminant flux rate per time (i.e., mass/area/time), most commonly reported as • g/min•m^2. Primary limitation of this technique is the lack of analytical sensitivity of the gas mixture due to the dilution from the injected inert gas.

C.8.3.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified in the HTRW technical planning process. The sampling strategy developed at a particular site will vary based on the intended purpose of the flux emission data. Flux chamber sampling strategy must address the VOC source, most likely route of travel, potential and real receptors affected by the VOC migration, and any target receptors with unique characteristics. Sampling strategies are based on subdividing areas into zones, and then sampling within each zone to assess zone variability. Many flux chamber applications and sampling strategies are either based on judgmental decisions or biased to site-specific conditions. For example, the effect of VOCs in creating an indoor air problem may necessitate the sampling of specific basement floor fractures and cracks. Also affecting the sampling strategy are the varying effects of changing meteorological conditions. The sample collection method must preserve the project concept of a representative sample. This may include maintaining sufficient duration for real-time measurements or obtaining a sufficient number of grab samples or a sufficient number of sample aliquots for compositing of time-integrated samples.

C.8.3.3 Sampling locations. Sampling locations will be determined by both the site-specific characteristics and the nature of the contaminants of concern. For indoor air issues, the sampling locations will likely be selected based on the receptors (buildings/structures/occupants) of concern. For emission flux data to be used for risk assessment or site-specific emission modeling, sampling locations should be located based on known or suspected contaminant sources and the most likely route of migration. For instance, migration may be determined through soil or concrete slab fractures, fuel emissions based on the known location of an underground storage tank or transport pipe, landfill cover deficiencies or openings, specific areas of a lagoon system, etc.

C.8.3.4 Sampling techniques and sample types. Figure C-25 shows a schematic of a flux chamber apparatus. A similar device that enables the user to sweep inert gas continually over a surface area and collect liberated volatile compounds from the isolated head space may be used. The specific application and contaminants of concern will usually dictate the design features of the sampler. Design parameters address the relative volume of the head space of the steady-state flux chamber and any special features for flotation, such as when evaluating a liquid source area. The volatiles swept from the flux chamber headspace can be monitored directly using field instruments or measurement devices, a sample can be collected from a series of grab samples and analyzed onsite, or a sample can be collected as aliquots and composited to constitute a time-integrated sample. In general, the liquid or solid surface is selected and prepared for the flux chamber application, as necessary. The characteristics of the sampling equipment and procedures are determined by the source media, the contaminants of concern, the detection limit measurement objectives, and the method of sample collection or detection. Flux chambers utilize an inert sweep gas from compressed cylinders of nitrogen or "zero air," or a purification system established for use with ambient air. For the latter approach, the effectiveness of the cleaning step should be verified. Refer to ASTM D 5314 for additional guidance on this type of sampling.

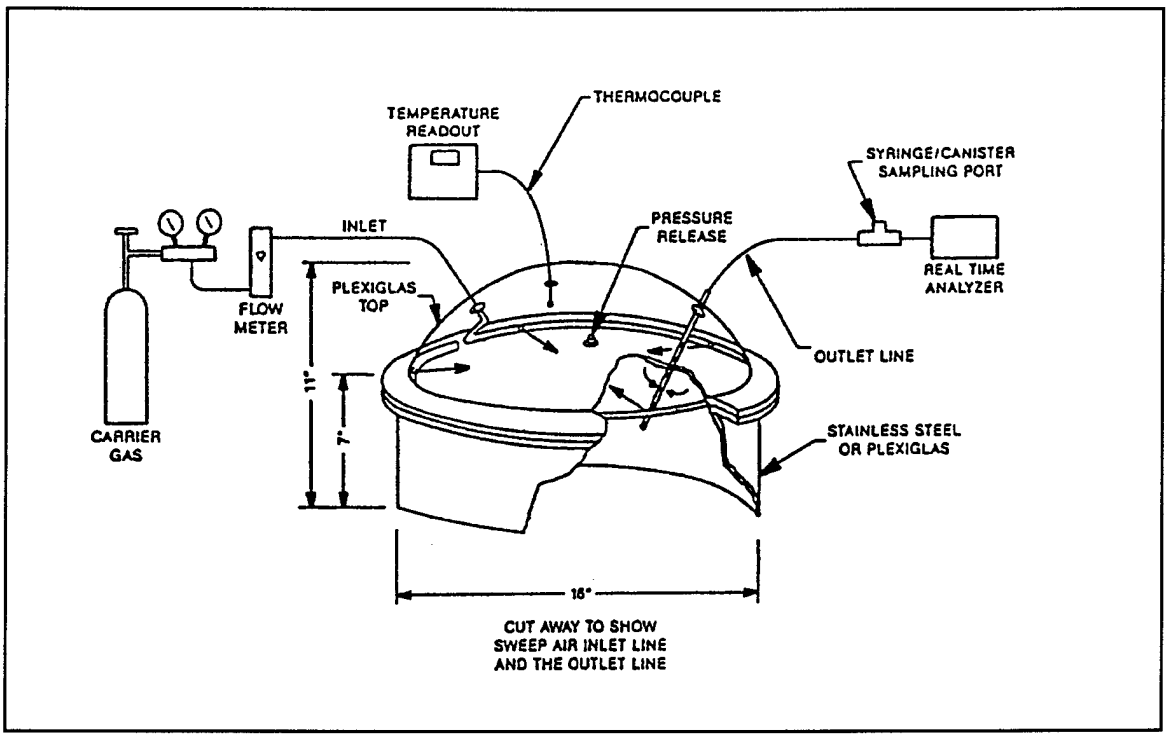


Figure C-25. Flux chamber apparatus

C.8.3.5 Sample preservation and handling. Methods for preservation of whole air and sorbent samples are similar to those identified for soil gas samples (Section C.8.2.4). Preservation of some absorption media samples may include cooling to 4°C ± 2°C to avoid contaminant loss or degradation. Glass collection cartridges may also be packed in metal containers to avoid breakage and photodegradation of contaminants.

C.8.3.6 Collection of adequate field control samples. Field control samples are collected to verify the integrity of the sampling system as interference-free, to evaluate reproducibility of the sampling procedures, and to determine whether the data are of acceptable quality. Field control samples include a variety of blanks, system checks, and the acquisition of replicate samples.

C.8.3.6.1 A system check should be done that isolates the flux chamber from the emission surface area, usually by inserting a sheet of inert and impermeable material on top of the surface area during a flux rate determination. This is also considered a field method blank. For whole air samples, method blanks should include collection of the sweep gas directly into a flexible or rigid container. This blank verifies sweep gas integrity.

C.8.3.6.2 For surface emission sampling, the flux chamber effluent may be split to collect duplicate whole air samples or duplicate media absorption to evaluate method precision. These samples may be composited from multiple grab aliquots, or successive grab samples taken by an emission extraction tee tube. For media absorption duplicates or split whole air samples, the flow rate and sampling times for each individual aliquot collected must be accurately measured and recorded. Audit gas standards can also be used

to evaluate system measurement performance but can add significant costs to the sampling and analysis program.

C.8.3.7 Sampling methods. Several varieties of devices may be configured to obtain a surface emission measurement. Time-integrated samples are collected for subsequent contaminant identification and quantitation using sorption media or whole air samples as discussed in Section C.8.2.4.1. Based on the sampler design, specifications must be established for its use. This includes identifying all sampling and support equipment necessary; establishing SOPs for sample collection; identifying the type of samples and sample containers used; determining the type, rate, and total volume of sweep gas; and determining the volume of emissions needed to meet the detection limit requirements. Such samples can be analyzed onsite or transported offsite for analyses. The utility of whole air samples is that one sample can be analyzed in duplicate, or by using separate aliquots, several different analytical procedures for different parameters or classes of compounds can be determined. For example, a gaseous landfill emissions whole air sample is typically analyzed for chlorinated and nonchlorinated VOCs, methane, sulfur gases and atmospheric "balance gases" including O_2, CO, and CO_2. Samples may also be collected on sorption media, such as Tenax, XAD-II, etc. Refer to the appropriate EPA Compendium Methods (EPA/600/4-84/041, EPA/625/R-96/010a, and EPA/625/R-96/010b) to determine the absorption medium needed. Collection of sorbent medium samples offers the ability to concentrate contaminants of concern to achieve a lower detection limit or long-term integrated sampling. For projects for which absorption media is selected as the sampling approach, recommend using the EPA Compendium Method TO-17 (EPA/625/R-96/010b) absorbance media evaluation and performance verification procedures.

C.8.3.8 Decontamination procedures. Requirements for decontamination of flux chambers are limited to specific applications such as after initial installation or if the chamber is moved from a potentially high concentration source area to a lower concentration or a trace-level source area. During field use, decontamination is usually limited to flushing the chamber with the sweep gas, either by placing the chamber on an inert surface or after placing the chamber at the measurement location and flushing to a steady-state emission concentration. When more thorough decontamination is necessary, follow procedures established in Instruction E-6 of Appendix E.

C.8.3.9 Documentation requirements. Documentation requirements are based on the information needed to support the final emission flux calculations. Critical field data are needed from both the site and sample perspectives. Record barometric readings during sampling events. This may be accomplished with an onsite barometer or from monitoring a local National Weather Station. Also record the temperature of the source matrix. The specifications should be used to determine the flux chamber dimensions. Note that any sampler-to-surface gaskets or sealants may alter the actual area of the flux. Document any carrier and calibration gases information, including cylinder serial number, contaminant and balance gases used, contaminant concentrations and any error associated with that concentration, cylinder gas pressure, cylinder receipt and expiration date, vendor providing the cylinder gas, a description of regulator gas pressure, gas flow rate measurement devices, and gas flow rate. It is highly recommended that the gas cylinder pressure be recorded during use to document the pressure decrease relative to time usage. Some contaminant concentrations become unpredictable when cylinder pressures drop below a certain pressure. Additional information needed may be referenced from the appropriate EPA or ASTM method.

C.8.4 Point source emissions and process sampling.

C.8.4.1 Scope and application. Point source sampling for HTRW projects is normally performed due to regulatory requirements to evaluate a remediation process, or to characterize a contaminant concentration for remediation feasibility, or system design and optimization. This type of sampling includes

the collection of gaseous phase or particulate matter (PM) and PM-related compounds. Regulatory emission compliance testing will involve specified USEPA sampling and analysis procedures. System process measurements are generated to evaluate efficiency of a recovery process, to determine operating parameters during system startup, or to verify the performance requirements of an Air Pollution Control system. These system measurements should utilize the most cost-effective method that produces data of acceptable quality for the defined use. Point source emissions gas phase sampling may also be required to evaluate emissions from a localized surface area that is directed through a single exit point or a vent system.

C.8.4.2 Sampling strategies. For point source emission sampling to meet compliance requirements, the regulation and specified method should be used to define the sampling strategy. To monitor system performance or contaminants associated with a remediation process, the sampling strategy should address the most representative sample extraction points for the inlet and outlet sample waste streams. The sampling strategy for measurements involving particulate-related contaminants must address sampling inhomogeneity, particulate stratification, and sampling velocity rate to satisfy the required gaseous/particulate separation and to comply with the measurement definition of particulate matter. Sampling for PM and PM-related compounds requires sample acquisition at an "isokinetic" sampling rate. Isokinetic sampling rate is defined as the collection of a gas/particulate sample through a tapered inlet sampling nozzle at the same velocity as the sample stream.

C.8.4.3 Sampling locations. Sampling locations for compliance monitoring are defined within the appropriate method, and address both the extraction point of the sample, relative to system design, as well as the total number of extraction points. For process waste stream sampling, the sampling location will be dictated by the type of information needed, the part of the system being evaluated, and the limitations of available sampling locations. Most sampling locations and sampling points are performed according to USEPA methodologies.

C.8.4.4 Sampling techniques and sample types. Most gas phase samples and particulate matter measurements are performed using extractive sampling procedures. The sampling techniques must address representative sample acquisition, sample transport and integrity, sample conditioning (when applicable), and sample contaminant separation and collection. For the majority of such measurements, the sample is extracted from the source and pulled through a sampling train with the use of a vacuum pump. The sampling train will separate particulate matter from the gaseous contaminants. Collection of the gaseous contaminants relies on either the capture on a sorbent (resin) media or in a liquid impinger absorption. Figure C-26 depicts a typical extractive sampling system including the extraction probe, the filter, heating device, postfilter volatile contaminant collection media, and condenser (impinger) absorption for collection of other contaminants.

C.8.4.4.1 Gas phase contaminants. Point source sampling systems designed to collect VOCs can either involve a PM system or function as a gas-phase-only type of collection. Systems established to collect VOC exclusively should remove and discard any particulate matter from the gas stream using a filtration procedure that minimizes method interferences. The filtration methods may include the use of a filter as depicted in Figure C-23 or use of a glass/quartz wool plug placed at the inlet of the sampling probe. For VOC sampling, heating the extraction probe and filtration devices is not necessary unless moisture condensation will result in a method interference. The gas phase contaminants may be collected on a sorbent medium, within a specified solvent, or by collection of a whole-air sample. Methods for the determination of VOCs can be found in 40 CFR Parts 60, 61, and 266 and EPA/SW-846, Chapter 10.

C.8.4.4.2 Particulate matter (PM) and PM-related compounds. Particulate matter samples and associated data can involve determining the gravimetric mass of PM collected for a given sample volume,

or the subsequent chemical analysis of that collected PM. Most point source sampling systems are designed to collect the PM in the "front half" of the collection system, followed by the collection of semivolatile organic compounds (SVOCs) and/or metals in absorption media. Figure C-26 depicts the extraction probe followed by the temperature-controlled filter. In terms of PM, the recovery of material from the method-specified collection points from the sampling probe assembly and the front half of the filter assembly, along with the material captured on the filter, is defined as PM. The PM adhering to the surfaces of the sampling nozzle, probe liner, and filter holder front half are recovered by brushing and then rinsing the PM into a collection vessel using an appropriate solvent such as acetone, methylene chloride, toluene, distilled water, etc. The mass of the PM is determined gravimetrically after solvent evaporation by subtracting the tare weight of the filter from the final weight of the sample and filter. For semivolatiles and some metals, it is necessary to collect and analyze both the collected PM and additional parts of the collection train.

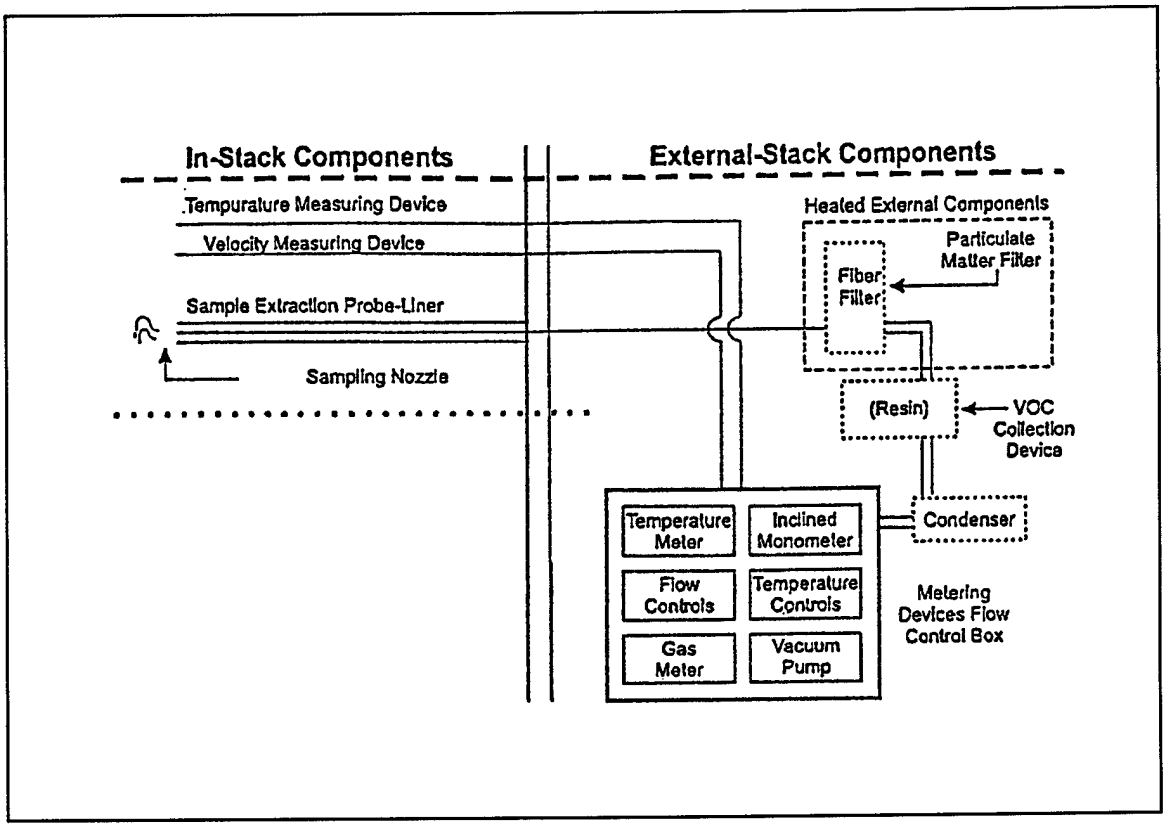


Figure C-26. Extractive sampling system

- The determination of SVOC is accomplished by recovering the probe and filter particulate and those SVOCs collected in the postfilter collection media. Commonly used collection media include Tenax and XAD-II resins. The PM is extracted using an appropriate solvent. Separately, or combined with the PM, the collection media is also extracted and the resulting extract is analyzed for the contaminants of concern. SVOC classes for HTRW projects include PCBs, dioxins/furans, PAHs, and project-specific SVOCs. The determination of both PM and SVOC can be accomplished using a single sample collection. However, it must be verified that the PM gravimetric weighing conditions specified by the method do not compromise the sample by loss

of other target analytes. Methods for the collection and analysis for these SVOCs are included in 40 CFR Parts 60 and in EPA/SW-846, Chapter 10.

- The determination of metals of interest is accomplished by recovering the probe and filter particulate and metals collected in the postfilter collection system. The metals collection system subsequent to the filter usually consists of acidic impinger solutions. When PM and metals are to be determined, the organic collection resins cannot be used in the sampling train. Methods for the determination of metals in point source emissions or process streams are included in 40 CFR Parts 60 and 266, and EPA/SW-846, Chapter 10.

C.8.4.4.3 Instrumental measurements. Most instrumental measurements of point source emissions are performed using Continuous Emission Monitors (CEMs). Applications to USEPA emission regulations require the use of CEMs. However, an equivalency program has been established to allow the use of alternative instrumental measurements proven equivalent to the regulatory specified standard. Gas phase measurements performed for purposes other than regulatory compliance may be accomplished by other means, so that the measurement DQOs are met.

C.8.4.5 Sample preservation and handling. Methods for preservation of process or point source emission samples include temperature and environmental control by method-specified procedures. In general, sample preservation of cooling to 4°C ± 2°C is recommended for organic samples. Absorption cartridges should be covered with clean aluminum foil for sample holding and shipment for analysis. Collection media such as filters and impingers do not typically require preservation except when noted within the method.

C.8.4.6 Collection of adequate field control samples. Field control samples are collected by the sampling team as specified by the project. Most sampling methods specify collecting reagent blanks and conducting field blank recovery. A method blank is collected by assembling the entire train as for sampling and recovering the sampling train components using method procedures. For point source sampling, duplicate samples are not routinely taken. This adds considerable time and cost to the project and is seldom required from a regulatory standpoint. Acquisition of duplicate samples for PM and PM-related parameters is also discouraged due to the significant difficulty in collecting these samples.

C.8.4.7 Point source or process sampling methods. Sampling methods are categorized based on the subsequent analyses. Unique sampling protocols are used for the acquisition of volatile contaminants, particulate matter, semivolatile and metals contaminant/particulate-related matter and physical parameters. Point source compliance test methods have been codified in four 40 CFR references: Part 51, Appendix M, Part 60, Appendix A, Part 61, Appendix B, and Part 266, IX. Point source sampling done to support HTRW projects has also been included within the most recent update of Chapter 10 of EPA/SW-846. Information on these methods can be utilized by accessing the USEPA Technology Transfer Network located at www.epa.gov/ttn and using the Emission Measurement Center technical site. Other testing groups, i.e., ASTM, have issued similar test methods that may be considered alternative sampling methods. Gas phase contaminants and physical characteristics are often measured by field instruments when long-term sample monitoring or a high number of sample measurements are required. Instrumental methods available for gas phase measurements are typically CEMs. Performance specifications, which qualify the applicability of the selected CEM to an emission, are found in 40 CFR Parts 60 and 266. Technical direction and operating performance requirements for CEMs used for operational monitoring can be found in 40 CFR Part 60, Appendix F.

C.8.4.8 Decontamination procedures. For most point source emission and process streams sampling, sampling train components should be thoroughly cleaned in a laboratory environment, using prescribed or project-defined protocols.

C.8.4.9 Documentation requirements. For all point source emission and process stream sampling methods the documentation of a considerable amount of sampling data is required. Methods for PM and PM-related contaminants require the periodic recording of operational data and sampling train parameters. Refer to National Source Performance Standards 40 CFR 60 (NSPS) Method 5 and other PM-related methods to provide field sampling data sheets outlining the required parameters to be recorded.

C.8.5 Ambient air sampling.

C.8.5.1 Scope and application. Ambient air measurement for HTRW projects can involve onsite, site perimeter, or offsite sampling locations. The measurements may involve particulate matter, gaseous contaminants, or meteorological parameters. With the exception of meteorological monitoring, onsite air measurements are typically performed for worker health and safety purposes, and the requirements are specified in the project site safety and health plan. USACE guidance to determine the applicability of this type of sampling may be referenced from EP 1110-1-21. The site perimeter and offsite air measurements are typically performed for regulatory compliance, as a result of an Air Pathway Analysis, to assure nearby public health and safety, to confirm effectiveness of process and onsite emission controls, and to comply with project requirements.

C.8.5.2 Sampling strategies. Sampling strategies are developed by the project team to satisfy project-specific data needs that are identified in the HTRW technical planning process. The sampling strategy developed at a particular site must include permanent and mobile sampling site collections, coordination of sampling locations with meteorological conditions and site activities, contingency sampling based on survey or optimized ambient air monitoring, and sampling based on site-specific sensitive receptors, terrain features, etc. Sampling strategies are often limited by site-specific characteristics and activities that vary during the project. Refer to EM 200-1-5 or EPA/451/R-93/007 for information on the development of ambient air monitoring sampling strategies.

C.8.5.3 Sampling locations. Sampling locations for site perimeter or offsite ambient air sampling are dependent on project data objectives. In the simplest form of perimeter monitoring, a single sample at a downwind location would require periodic sampling during pertinent activities. If the objectives include calculating a net site contribution, then a corresponding upwind sample will be required. If short-term (less than 24-hour) sampling is required, a mobile sampling apparatus that is moved to accommodate the changing downwind location or a minimum of four permanently established sampling locations which are operated only periodically will be necessary. Greater limitations are usually experienced for offsite sampling locations and frequencies than at the perimeter due to location accessability, power supplies, and neighborhood acceptability and environments. For some longer term HTRW projects a real-time perimeter monitoring system may be most feasible. An alternative "sector monitoring" sampling strategy may be applicable. Offsite sample locations may pose additional difficulties due to location accessability, power availability, and community acceptance.

C.8.5.4 Sampling techniques and sample types. A variety of sampling and measurement techniques are available that address ambient air monitoring at HTRW project sites for general classes of VOCs, SVOCs, particulate matter, metals, and meteorological parameters.

C.8.5.4.1 Gas phase contaminants. Gaseous ambient air contaminants can be measured directly with onsite instrumentation or by sample collection and analyses. Collection of gaseous ambient air VOC components of interest is typically accomplished by collecting a "whole-air" sample within a flexible or rigid container or by absorption of the compounds within a collection resin. Figure C-22 depicts a summa canister, which has an internal surface that maintains sample component stability. Figure C-23 depicts an absorption resin collection cartridge, which collects and concentrates the VOCs of interest. Several of the Compendium TO-methods specifically address VOCs including TO-1, -2, and -14 (EPA/600/4-84/041), -16, and -17 (EPA/625/R-96/010b). These methods should be selected to satisfy project-specific components of interest, the applicability of the method to site-specific conditions, and the ability of the method to provide project-required data quality objectives.

C.8.5.4.2 Particulate matter (PM) and related compounds. The collection of PM in ambient air is typically accomplished by passing the sample through a preweighed filter. The filter is subsequently reweighed for a quantitative determination of the PM. PM in ambient air is categorized as total suspended particulate or some size fractionation. These fractionations range from a PM less than 10 microns (PM-10) to a lesser size such as PM-2.5. To determine the appropriate categories, the PM-10 or PM-2.5 is quantified gravimetrically as mass per volume after separation from the larger total PM. Similar to point source PM measurements, ambient air PM can be collected to determine SVOCs or metals. A typical total suspended particulate matter collection device is depicted in Figure C-27.

- SVOCs sampling includes the collected PM fraction of the sample, and the fraction that passes through a filter media and is collected on an appropriate sorption medium. Most SVOC collections are performed using a filter followed by a sorbent media of polyurethane foam or XAD-II resin. The SVOC classes often required for HTRW projects include PAHs, pesticides, PCBs, dioxins/furans, etc. When projects require more than one of these SVOC classes, recommend that procedures for sample collection, recovery, preparation, and analysis be combined. Also recommend that project-specific SOPs be generated to assure that the procedures meet project sensitivity and QC requirements.

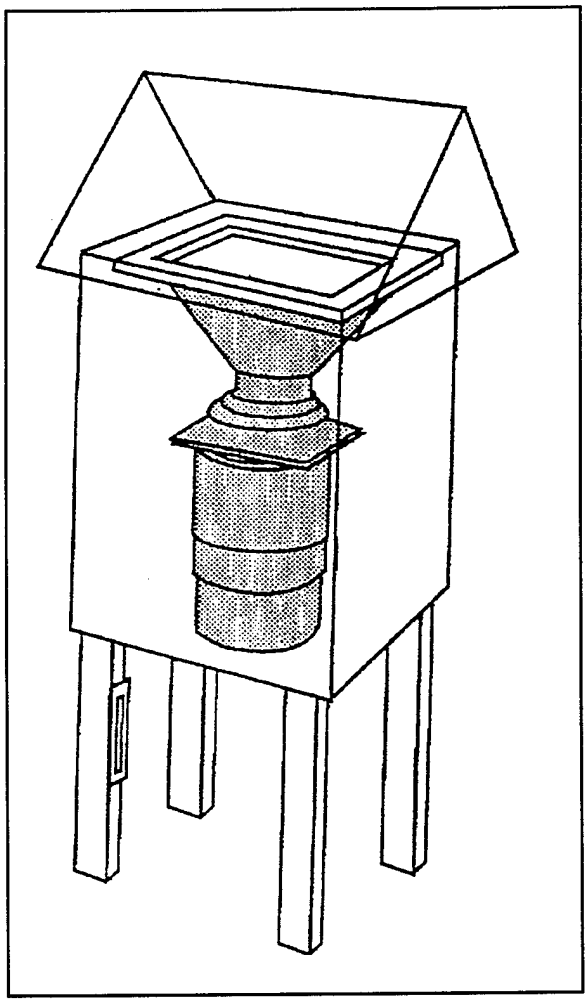


Figure C-27. Total suspended particulate matter sampling system

- Sampling and analysis for metals in ambient air are usually accomplished by using the total suspended particulate collected sample after reweighing. A fraction of the collected filter/PM is digested for the appropriate analytical procedure. The selection of the collection filter media must be performed with care relative to the required metal measurement method detection limit and the method-required filter blank correction. Factors including the metals of concern and the amounts of their presence in the blank filter are crucial to filter media selection.

C.8.5.4.3 Instrumental measurements. A variety of ambient air instrumentation and measurement technologies have been developed to support the USEPA ambient air programs and serve as criteria pollutant monitors. Many of these vendor instruments and monitors have been granted equivalency status by USEPA to monitor for criteria pollutants such as nitrogen dioxide (NO_2), sulfur dioxide (SO_2), ozone (O_3), and PM-10. These instruments have met the equivalency criteria set by USEPA and will generate equivalent results if performed following the specified operating requirements. For other ambient air sampling uses, the instrument selected for the measurement must meet the project DQOs.

C.8.5.5 Sample preservation and handling. Methods for preservation of whole air and sorbent samples range from the simplified canister, which is shipped at ambient conditions, to the enclosure and low temperature maintenance of recovered VOC collection cartridges. Factors to be considered for sample preservation include the sample contaminant loss and degradation during storage and shipment, sample media breakdown or breakthrough, etc. The EPA Compendium Methods (EPA/600/4-84/041, EPA/625/R-96/010b) may not identify the sample preservation necessary for all site specific samples or environments. Additional measures may be necessary, such as the storage of flexible VOC bags or collected resins within enclosed containers, or aluminum foil for protection from photodegradation.

C.8.5.6 Collection of adequate field control samples. Field control samples are collected to verify that the sampling system is interference-free and to determine whether the data are of acceptable quality. For ambient air sampling, recommend a variety of blanks be incorporated to evaluate the sampling and measurement system. These include evacuated canisters and resin collection media blanks, reagent sampling, and recovery blanks. When site conditions exist that may invalidate or jeopardize results, recommend the collection of backup samples (i.e., VOC) in series with the method media.

C.8.5.7 Sampling methods. There are seven codified USEPA methods (40 CFR Part 50), including total suspended particulates, carbon monoxide (CO), SO_2, O_3, NO_2, lead (Pb), and particulate matter less than 10 microns (PM-10). USEPA also allows the use of equivalent measurement methods for ambient air criteria pollutant monitoring. When these methods serve compliance monitoring, include quality control procedures and results to support the data quality required. The QC procedures outlined within several standard methods may serve as a guideline for air methods that do not specify appropriate QC procedures. Other EPA ambient air methods used for HTRW projects have been published as EPA guidance methods within USEPA (EPA/600/4-84/041, EPA/625/R-96/010b). These methods are referenced as the "TO" methods and include TO-1 through TO-17. For inorganic matter parameters that can be used for HTRW projects, the USEPA has published guidance methods (EPA/625/R-96/010a).

C.8.5.8 Decontamination procedures. Decontamination procedures for ambient air sampling applies to the sampling apparatus and the reuse of sample containers (i.e., summa canisters or flexible bags for whole air samples). Decontamination of apparatus is accomplished by flushing the equipment with a inert gas until the system is found to be interference-free. If flushing the system is ineffective, more thorough procedures for decontamination as identified in Instruction E-6 of Appendix E should be used. Decontamination of flexible bags may be accomplished by a series of inflate-and-deflate steps, using the inert gas until the filled

bag is analyzed and found to be contaminant-free. Generally, filling and evacuating three successive times is taken to be acceptable. For any given site application, this should be confirmed.

C.8.5.9 Documentation requirements. Documentation requirements for ambient air sampling should encompass all aspects of the site setup and sample collection and handling as outlined within this instruction and in Instruction F-1, Appendix F. This includes documenting the sample equipment and apparatus preparatory phase inspection and quality check before field activities begin. Sampling equipment calibration may include volumetric measurement devices and flow controllers. Certification of VOC sampling systems include equipment certification. The cleanliness of each lot of precleaned sample containers or collection media should be verified by the container supplier or the laboratory, and appropriate paperwork (i.e., certificates) retained with other field documentation.

Appendix D
Hazardous Waste Sampling Instructions

D.1 Bulk Material Sampling

D.1.1 Scope and application. Instructions presented in this section are for collecting representative solid or liquid samples from various containment vessels. These include tanks, drums, waste piles, fiber drums, sacks, bags, and similar small containers. Sampling containerized materials can present unique obstacles to field personnel. Buried or underground containers, container staging, identification, opening, and sampling are all issues to be considered. Instructions for sampling containerized materials by the following techniques are included in this section: scoop or trowel, waste pile sampler, Veihmeyer sampler/corer, sampling trier, grain sampler, composite liquid waste sampler (COLIWASA), and an open tube sampler. The Bacon bomb sampler described in Instruction C-3 (Appendix C), can be used for sampling liquids from large storage tanks.

D.1.2 Sampling strategies. Sampling strategies must be geared toward the type of wastes anticipated to be encountered, whether they are above ground or buried, with an important emphasis placed on safety. Safety considerations include the safety of personnel conducting the work, the surrounding community, and the environment. Occupational Safety and Health Administration standards and regulations should be followed, and appropriate monitoring equipment should be used during sampling operations. Sampling strategies are also determined by the stage of the investigation. For instance, during a preliminary assessment of an inventory of containers, it is more appropriate to identify the number of containers present, and take a random sampling of the inventory to identify gross categorization of the waste inventory. The amount of sampling will depend upon the number of total containers and whether there are similar markings on the containers. This information may be used to estimate the cost of cleanup, including the costs involved with transportation, treatment, storage, and/or disposal of the wastes. Later stages of the investigation may require sampling and analysis of all the containers individually to identify the waste category and compatibility of the wastes if bulking operations are planned. Biased sampling techniques are typically used to sample containers.

D.1.2.1 Sampling locations. If buried containers (drums, tanks) are investigated, the location of the containers must be determined. Information should be obtained from historical records. Past areal photography of the area may also be helpful in identifying areas that have been filled in over time, show evidence of stressed vegetation, etc. Geophysical techniques may also be used to locate potential caches of containers. Actual test pit excavations may be made to verify other less conclusive evidence. For drums and other containers that may be staged, locations should be identified for sampling purposes. Typically these areas are temporarily bermed, with sufficient safety and spill cleanup equipment easily accessible. If few containers are encountered and the conditions are secure from a safety standpoint, sampling may be conducted in place. In any kind of container sampling, however, remote operations for handling, staging, and opening are recommended.

D.1.2.2 Types of samples. All waste samples must be grab samples. Until waste characterization and compatibility testing are completed and confirm that the wastes are compatible, containerized samples should never be composited. Composites are collected only just prior to or during bulking operations. Normally, a composite sample is acquired from the bulked waste in the same proportions of the original waste stream to verify the applicability of disposal options. It should be noted, however, that composite samples can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix. Therefore, initial waste characterization screening must be thorough enough to avoid this problem.

D.1.2.3 Suggested samplers and handling equipment. Each sampling technique presents various dis advantages and advantages for its application. For example, sample disturbance, sample volume, chemical/ physical reactivity between potential contaminants and sampling tool materials, and ease of use and decontami-

nation vary from technique to techniques. The advantages and disadvantages of each sampling technique are presented in the discussion of the technique. Specialized equipment is available for handling drums and other bulk containers. Backhoes equipped with nonsparking bucket teeth and plexiglass safety cab shields are available for excavating and handling drums and bulk containers. Other excavating and handling equipment include backhoes with a drum grappler, industrial vacuum loaders, drum lifting yokes, and metal hoists. Special tools used to manually open drums for sampling include bung wrenches, drum deheaders, hand picks, pickaxes, and hand spikes. Remote devices for opening containers include backhoe spikes, hydraulic drum openers, and pneumatic devices. It is emphasized that remote handling and opening techniques are recommended, especially if the contaminants are not known or when container integrity is poor.

D.1.3 Sample preservation and handling. Bulk samples are considered medium- to high-concentration samples and may not need to be preserved, except for cooling and/or protection from light in some cases. Special procedures and techniques for transporting the samples to the offsite laboratory may be necessary, i.e., secondary containment within a paint can or similar container, as discussed in Instruction F-2, "Packaging and Shipping Procedures," Appendix F.

D.1.3.1 Sample containers. As noted, bulk samples are considered medium- to high-concentration samples. Generally, the sample volumes needed for analyses are smaller than those needed for samples in an environmental media. Also, if similar chemical parameters (i.e., extractable organics) with the same preservation requirements are required by the project, these chemical parameters may be combined into one sample container to minimize the amount of concentrated waste sampled and shipped. When samples are collected from concentrated waste samples for analysis of metals, a 125-ml wide-mouth glass container with PTFE-lined polypropylene caps may be used (PTFE is commonly referred to using the registered name of Teflon). When organics are the analytes of interest, 125-ml wide-mouth glass jars for extractables and as prescribed within the analytical method for purgeable organics should be used. All containers must have PTFE-lined caps. Containers should be cleaned based on the analyte of interest. Instruction E-6, "Decontamination Procedures," contains additional information on appropriate glassware cleaning protocols. If precleaned bottles are used, the cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) retained with other field documentation. Analytical protocols for bulk samples typically focus on simple tests to characterize the waste, or encompass analytical parameters that define the disposal requirements or disposal options available. Chemical parameters may differ from routine environmental samples and the individual tests may be combined into fewer actual sample containers, but the collection order should still be performed in the order of the volatilization sensitivity of the parameters. A general collection order for some common parameters follows:

- Volatile organics (VOA) - total or toxicity characteristic leaching procedure (TCLP).
- Purgeable organic carbon (POC).
- Purgeable organic halogens (POX).
- Total organic halogens (TOX).
- Total organic carbon (TOC)
- Extractable organics - total or TCLP.
- Total metals - total or TCLP.
- Phenols.
- Cyanide - total or reactive.

- Sulfide - total or reactive.
- British thermal units (BTU) contents.
- Radionuclides.
- Ignitability.
- Corrosivity.
- Oxidizer test.
- Peroxide test.
- Density.

D.1.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and to reduce sorption effects. Preservation methods for samples from bulk containers are limited to cooling and protection from light.

D.1.3.3 Special precautions for bulk and container sampling. Bulk sampling typically involves sampling medium- to high-level contaminants. Consequently, special precautions are warranted. Prior to collecting samples, a sampling plan should be developed that includes the following: research about the waste; identification of the drums to be sampled; selection of appropriate drum opening and sampling device(s); determination of the number and volume of samples to be taken; the analytical protocols; and development of procedures for opening drums, sampling, sample packaging, and transportation. A phased approach is recommended when evaluating bulk containers. The investigation may include all or only parts of the phased approach: reconnaissance, staging, opening, sampling, and packaging.

D.1.3.3.1 A preliminary assessment of each drum shall be performed prior to opening or sampling activities. During this reconnaissance phase, visual observations are recorded of container conditions (integrity), any markings, composition of the container, and whether it is an open-top container or a closed-top (bung type) container. Any special problems, i.e., evidence of pressure buildup (bulging) or access problems, should be recorded. Also note any recommendations with respect to opening/sampling these unique situations. Ambient air monitoring (for explosive and organic vapors) and radiation monitoring should be performed concurrently with these activities. Special precautions should be implemented and segregation of any stainless steel or nickel containers and gas cylinders should be noted, due to the potential for highly reactive, toxic, pressurized, and/or shock-sensitive contents. Containers whose integrity is breached should be overpacked immediately, or as soon as possible, to prevent further contaminant migration. Any markings noted on the containers should be considered suspect and noted for informational purposes only.

D.1.3.3.2 All bulk container contents should be approached as if they are unknown. Drum staging may be required at sites that have a large number of drums. The purpose of drum staging is to respond to obvious problems that might impair worker safety; unstack and orient drums in a prepared area that minimizes the spread of contamination during opening and sampling; and if necessary, organize drums into different areas to facilitate characterization and remedial action. Handling may or may not be necessary, depending on how the drums are positioned at the site. Prior to handling the drums, all personnel should be warned about the hazards and instructed to minimize handling the drums as much as possible. In all phases of handling, personnel should be alert for new information about potential hazards and should respond to new hazards before continuing with routine handling operations. Sampling areas should be adequately prepared (i.e., lined with plastic sheeting) to minimize the spread of contamination. However, it may not be practical to line the entire area with plastic sheeting due to the use of heavy machinery in the movement of drums. Empty overpack drums. An adequate

volume of absorbent should be kept near areas where minor spills may occur. Where there is a potential for large spills, a containment berm should also be constructed around the area where opening and sampling of drums will occur. If drum contents spill, personnel trained in spill response should isolate and contain the spill. Unique drum sample identification numbers should be assigned and written on drums. For large sites, it is recommended that a grid be applied to the site, and information from the original drum area be incorporated into the drum sample identification number. This allows the drum sample identification number and associated results to include aspects of the drum origin and location.

D.1.3.3.3 Remedial and emergency operations may require a separate drum opening area. Procedures for opening drums are the same, regardless of where the drums are opened. Drum opening tools should be nonsparking in nature, and include both manual and remote types. Manual drum opening tools include a universal bung wrench, drum deheader, manual drum punch (pickaxe, hand pick, or hand spike) (Figures D-1, D-2, and D-3). Remote drum opening tools include a backhoe spike and a variety of hydraulic or pneumatic drum openers (Figures D-4 and D-5). Many drum openers damage the drum integrity, making final bulking of contents or overpacking necessary. The method of opening selected should be based on drum condition, accessibility, knowledge of drum contents, whether direct contact is allowed or remote contact is necessary, and if the integrity of the drum should be maintained. Immediately after the drum is opened, recommend continued air monitoring of the area around the drums for explosive and organic vapors.

D.1.3.3.4 Drum sampling can involve direct contact with unidentified wastes. A trained health and safety professional should determine the appropriate personal protection to be used during sampling, decontamination, and packaging of the sample. Worker safety should be maximized during drum opening and sampling activities. Initially, drums should be inspected to verify contents are deeper than 25.4 mm (1 in.). If contents are less than 25.4 mm (1 in.) deep, the drums should be considered empty and disposed of accordingly. Drums verified as containing • 25.4 mm (1 in.) should be sampled for waste characterization testing (compatibility) and compatibility testing as detailed in Section D.1.8 for segregation, bulking, and disposal determinations. Section D.1.4 introduces several techniques that may be used to sample drum contents. Samples should be taken from each drum in sufficient volume and labeled with the assigned drum sample number, and information recorded in the field logbook or drum log sheets. Hazardous characterization (HAZCAT) and compatibility testing may be performed onsite or offsite at a laboratory.

D.1.3.3.5 Based on hazardous characterization and compatibility testing results, the waste drums should be removed from the staged sampling area, segregated into compatible waste drum groupings, and a composite sample of the compatible materials prepared for disposal analyses defined by the treatment, storage, and disposal (TSD) facility. All incompatible wastes should be overpacked if integrity is breached, and segregated for offsite disposal. Compatible drum contents may then be physically bulked, or the individual drums prepared for offsite shipment to a TSD facility.

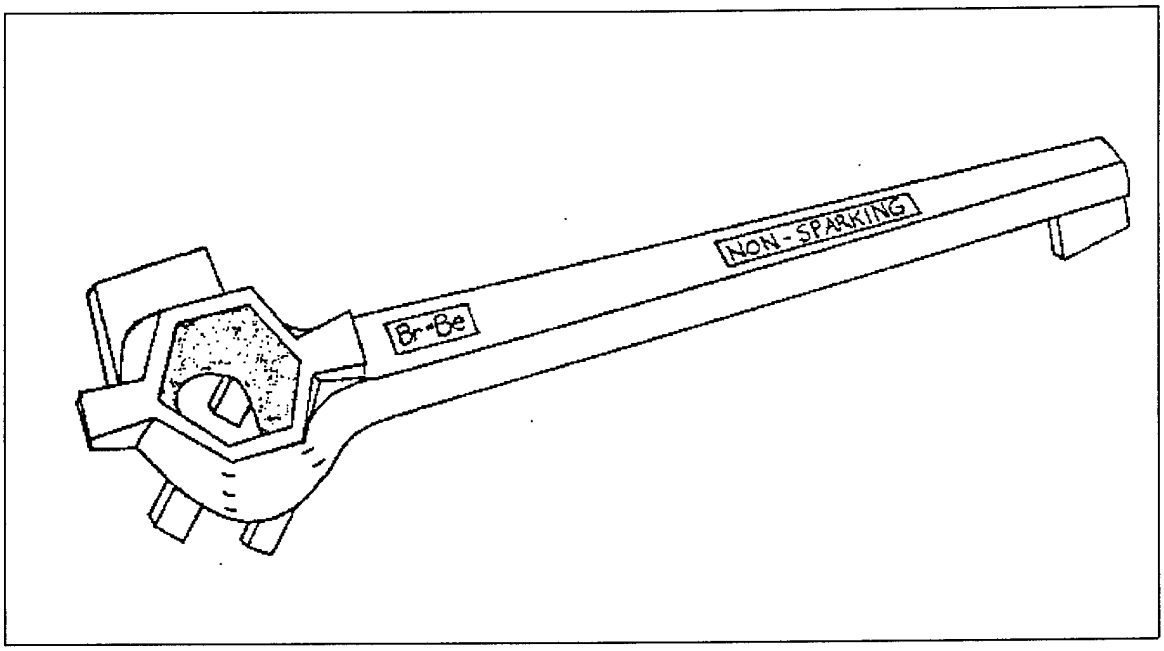


Figure D-1. Universal bung wrench

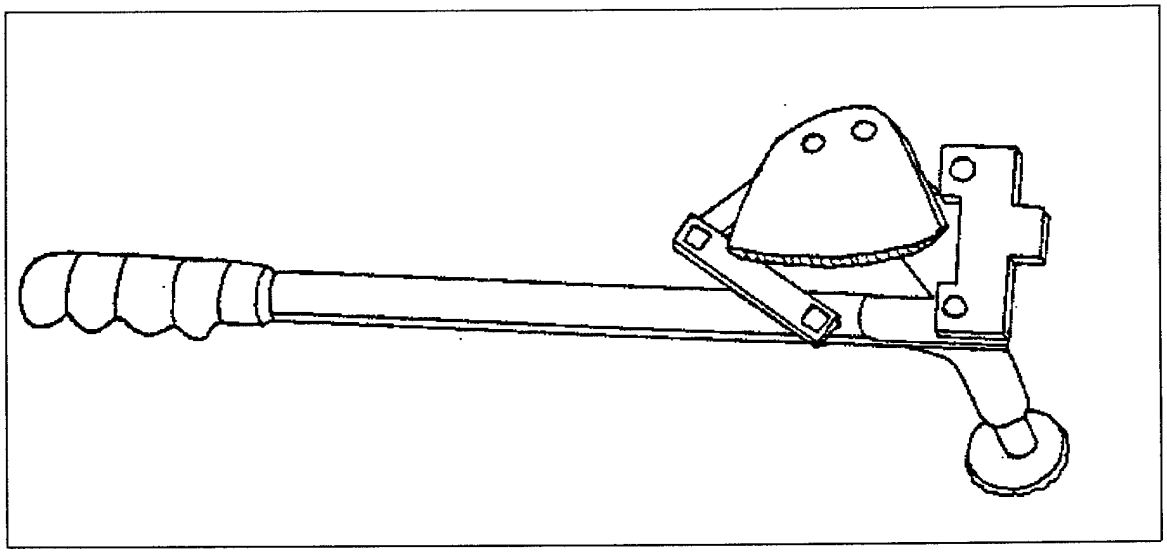


Figure D-2. Drum deheader

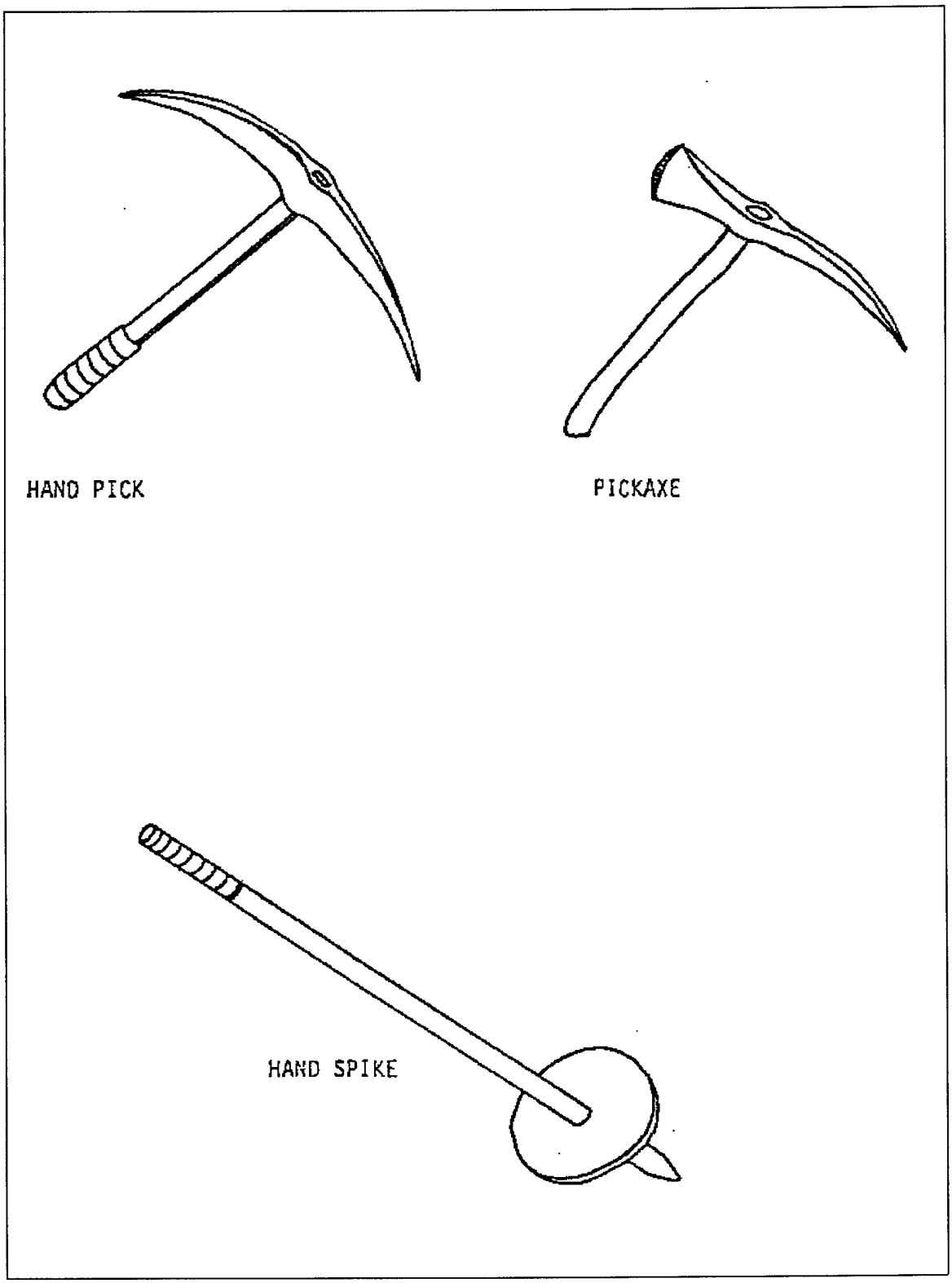


Figure D-3. Manual drum opening tools

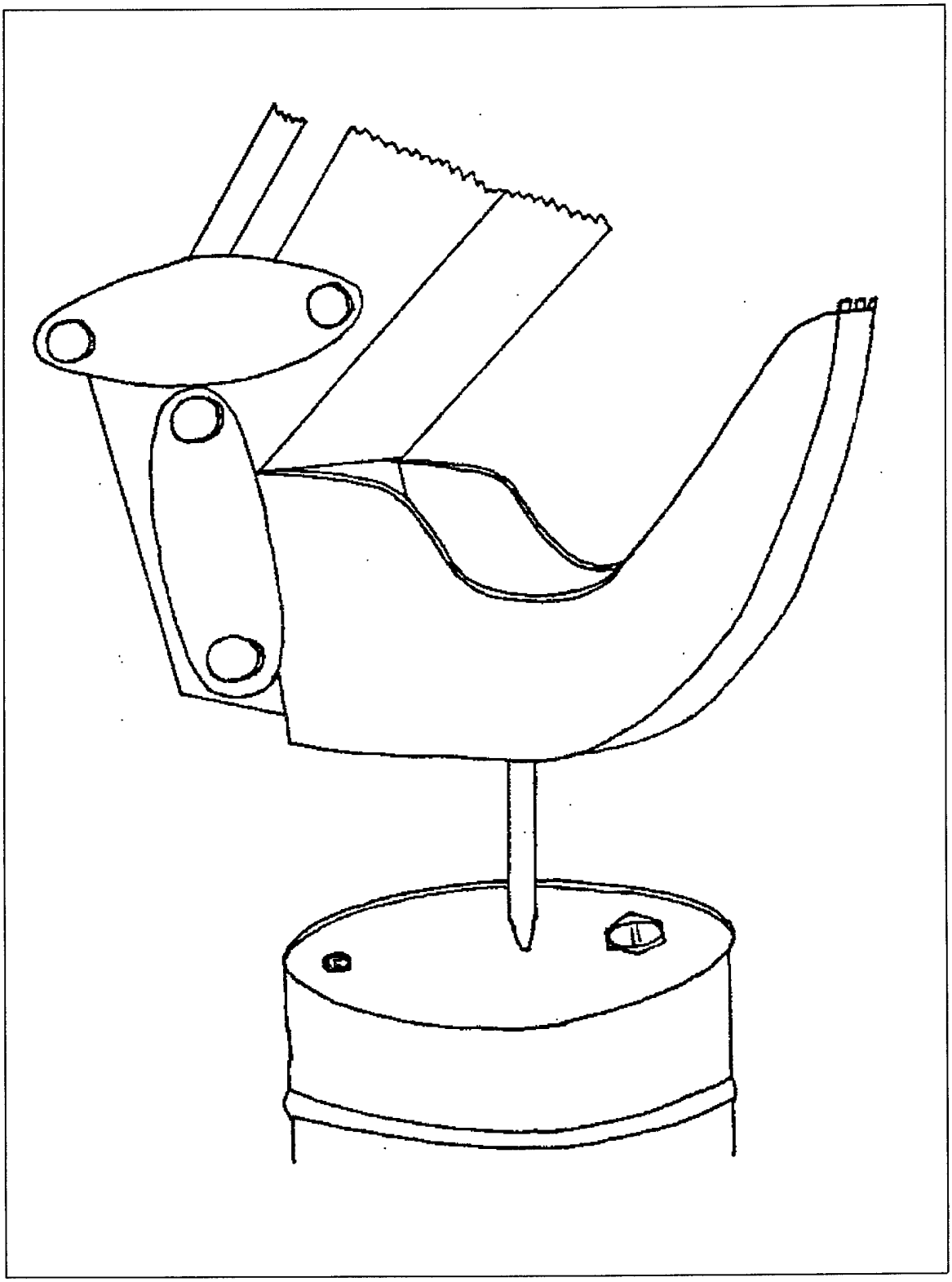


Figure D-4. Backhoe spike

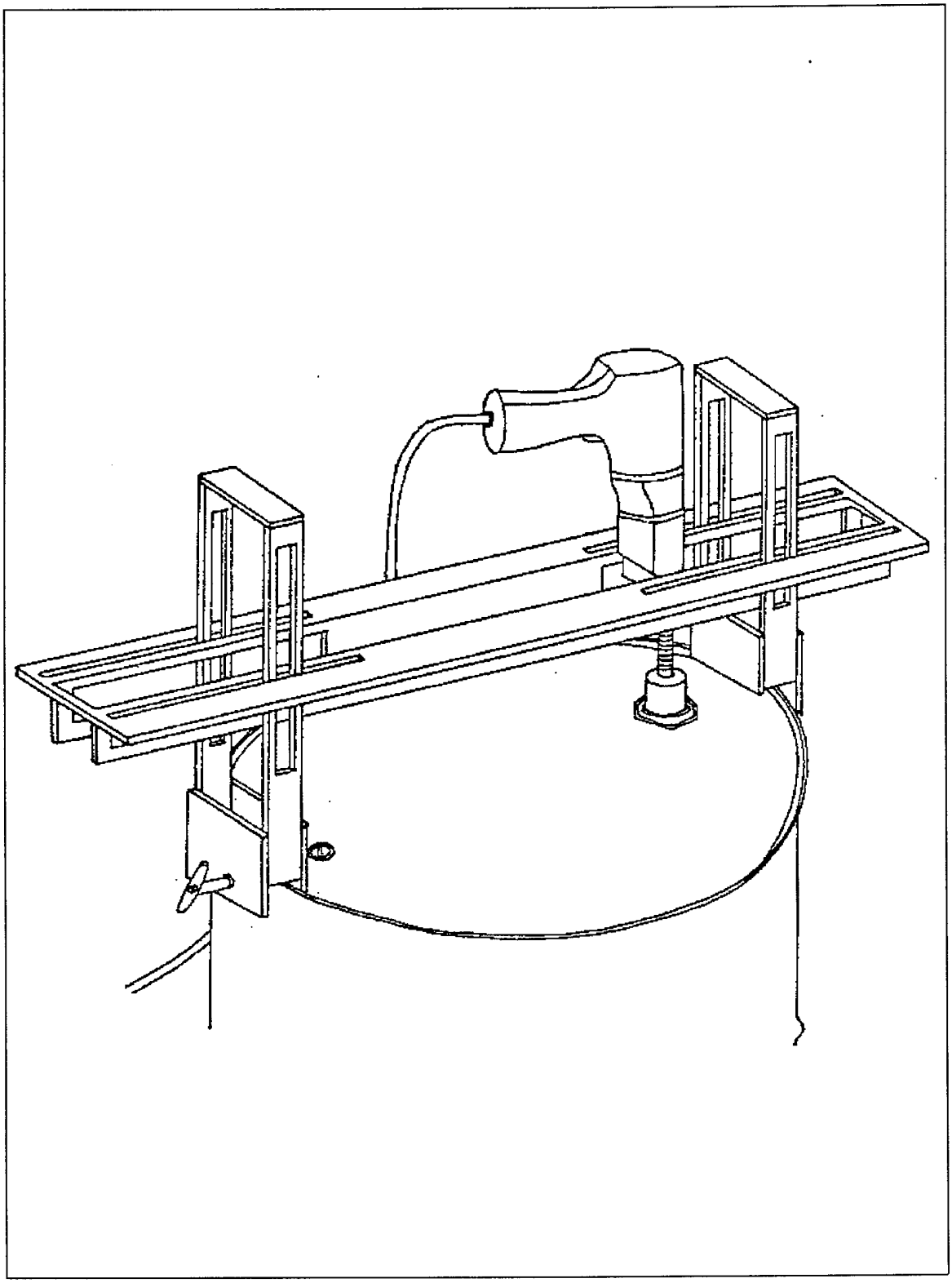


Figure D-5. Pneumatic drum opener

D.1.4 Sampling methods. Sampling instructions for the most common techniques of collecting liquid and solids samples from containers are as follows.

D.1.4.1 Open-ended tube. Method References: American Society for Testing and Materials (ASTM) D 5743 and EPA/540/P-91/008 (Standard Operating Procedure (SOP) #2009).

D.1.4.1.1 Applicability. The method provides a quick, relatively inexpensive means of collecting concentrated wastes. After sampling, the tubing can be discarded, thus eliminating the need for decontamination and the potential for cross-contamination.

D.1.4.1.2 Method summary and equipment. Liquid samples from open containers can be readily collected by merely submerging lengths of tubing into the containers (Figure D-6).

D.1.4.1.3 Sampling procedure.

- Insert tubing slowly almost to the bottom of the container. Approximately 0.3 m (1 ft) of tubing should extend above the drum. Use the tube to measure any sludge in the bottom of the drum.
- Be sure that the liquid in the container maintains a constant level in the tube during the descent of the tube.
- Cap the top of the sampling tube with a tapered stopper or gloved thumb, ensuring that the liquid does not come in contact with the stopper or thumb.
- Carefully remove the capped tube from the container and insert the uncapped end into the sample container, being careful not to spill any liquid outside the container. Removal of the tube from the container may require a step or platform aid.
- Release the stopper or thumb and allow the liquid to drain into the sample container until the appropriate sample containers are filled. Repeat as necessary.
- Remove the tube from the sample container and dispose of properly.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents, drum log sheets, and/or record in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

D.1.4.2 Composite Liquid Waste Sampler (COLIWASA). Method References: ASTMs D 5495, D 5743, and EPA/540/P-91/008 (SOP #2009).

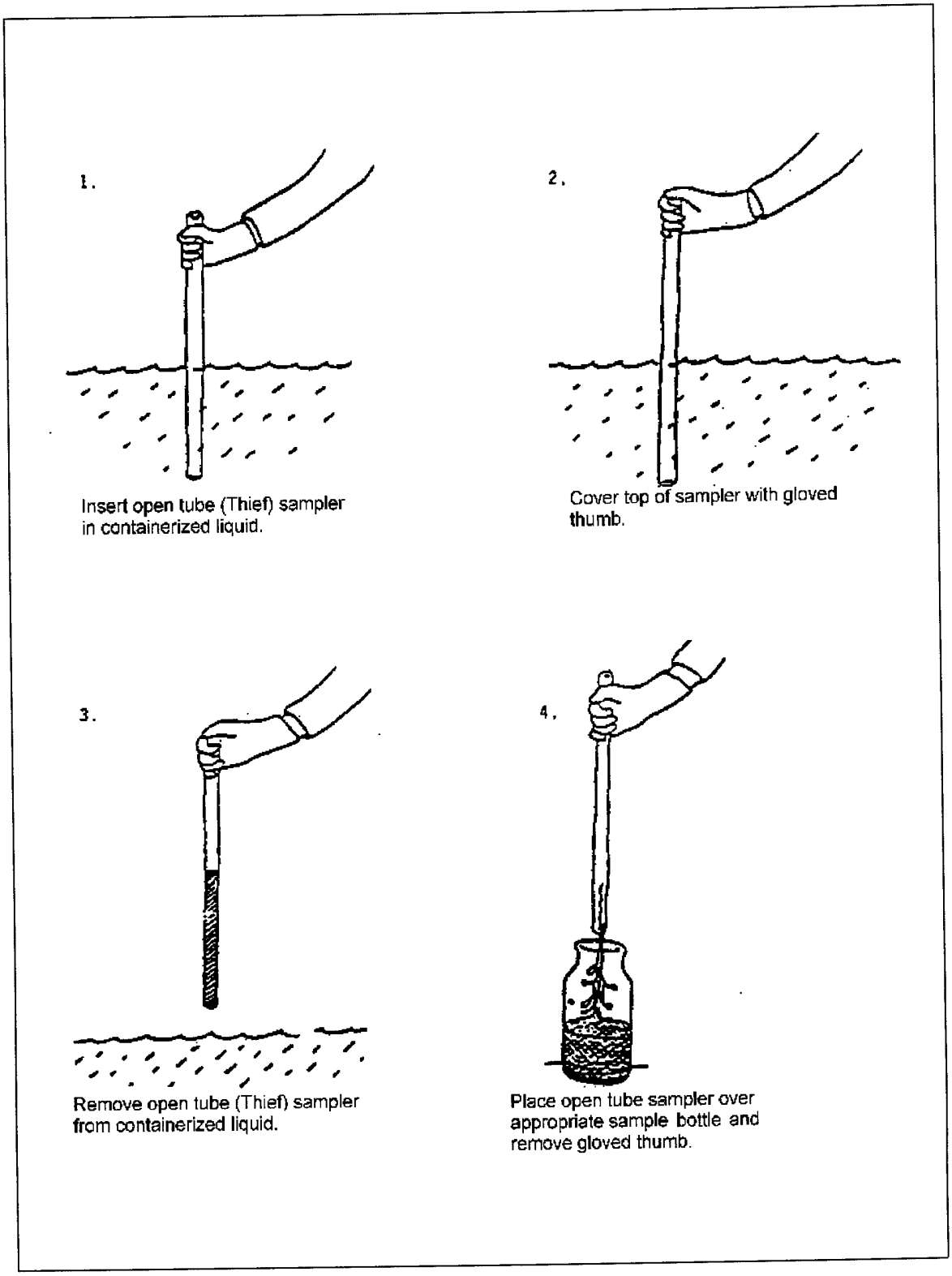


Figure D-6. Open-ended tube sampling procedure

D.1.4.2.1 Applicability. The COLIWASA permits the representative sampling of wastes having a wide range of viscosity, corrosivity, volatility, and solids content. Its simple design makes it easy to use and enables the rapid collection of samples, thus minimizing the exposure of the sample collector to potential hazards from the wastes.

D.1.4.2.2 Method summary and equipment. A properly constructed COLIWASA can be used to sample very hazardous materials safely and quickly.

D.1.4.2.3 Sampling procedure.

- Choose the plastic (Figure D-7) or glass ball type stopper (Figure D-8) COLIWASA for the liquid waste to be sampled and assemble the sampler.
- Ensure that the sampler is functioning properly. Adjust the locking mechanism if necessary to make sure the stopper provides a tight closure.
- Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the locking block of the sampler. The open position for the glass ball type is achieved by pulling up on the inner rod, thereby pulling the glass ball away from the tapered end of the outer tube.
- Slowly lower the sampler into the liquid waste. (Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same. If the level of the liquid in the sampler tube is lower than the level outside the sampler, the sampling rate is too fast and will result in a sample biased to bottom contents.)
- When the sampler stopper hits the bottom of the waste container, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the closed position by turning the T-handle until it is upright and one end rests tightly on the locking block. The closed position for the glass ball type is achieved by pushing the glass ball end of the inner rod against the tapered end of the outer tube.
- Slowly withdraw the sampler from the waste container with one hand while wiping the sampler tube with a disposable cloth or rag with the other hand.
- Carefully discharge the sample into a suitable sample container by slowly opening the sampler. This is done by slowly pulling the lower end of the T-handle away from the locking block and pulling up on the inner rod to release the contents while the lower end of the sampler is positioned in a sample container. Repeat as necessary.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents, drum log sheets, and/or record in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

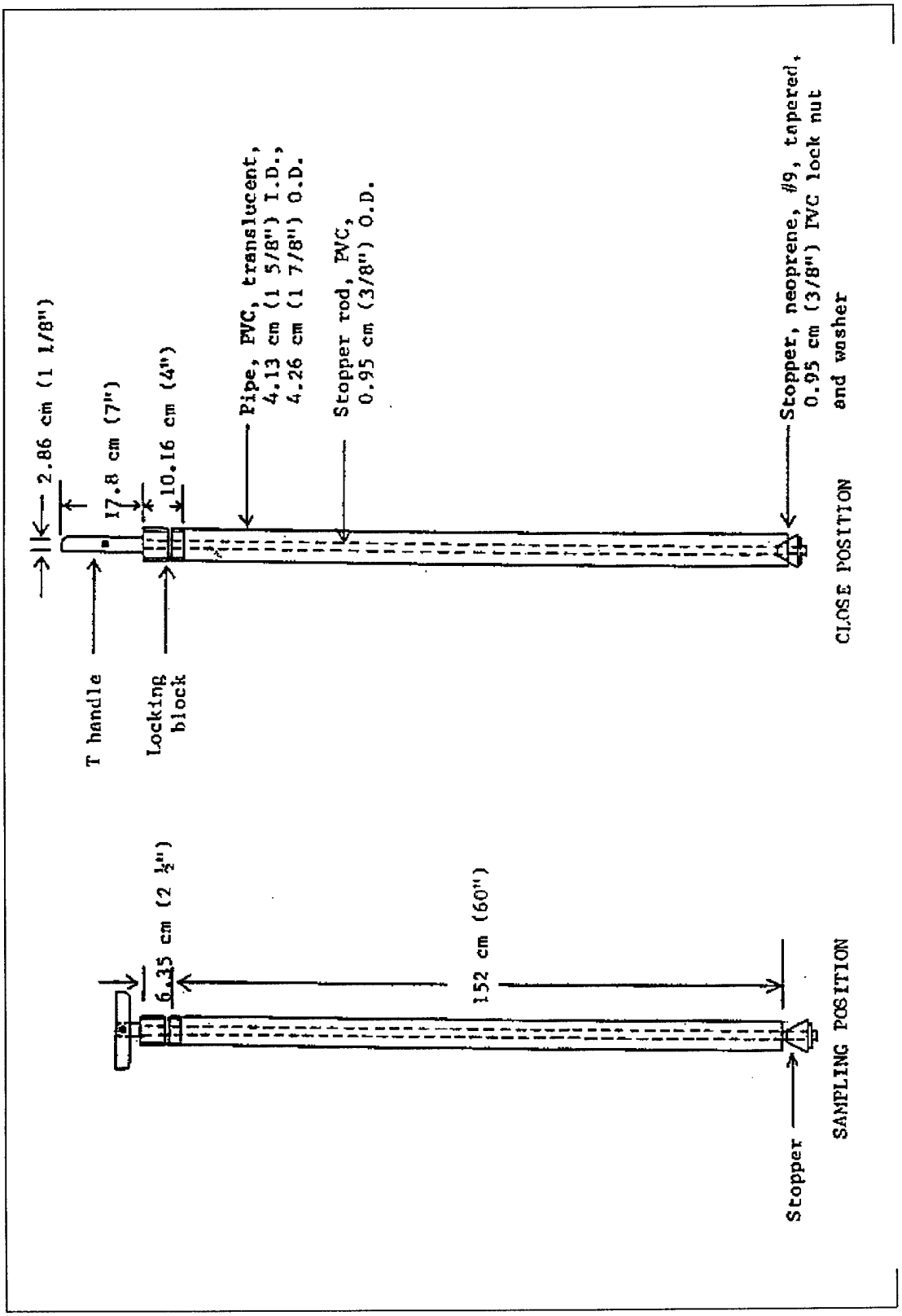


Figure D-7. Composite Liquid Waste Sampler (COLIWASA)

- Decontaminate the sampling equipment after use and between sampling locations. Since the tube is small and difficult to decontaminate, it may be cost-effective to dispose of the sampler and use new ones for additional sampling.

D.1.4.3 Emerging and innovative sampling procedures. Due to the difficulties with the use of a glass thief and COLIWASA sampler, recent modifications have been marketed to minimize spillage and sampling bias. The apparatus consists of a sample tube, an outer wiper for the sample tube, a head section that connects the sample tube directly to the sample container, and a plunger on a plastic line/rod or stainless steel rod that aids in the transfers of the waste to the sample container. The unique design minimizes spillage from several aspects, including the plunger assembly, which pulls the waste up the sampling tube into the head section and allows drainage into the sample container; the head section, which effectively connects the sample tube to the sample container, thereby completely enclosing the waste during this transfer; and the outer wiper, which cleans the outside of the sampler tube upon retrieval of the sampler. The features that isolate the waste will also minimize the potential for sampler exposure to the waste or cross-contamination of the samples. All components may be reused with decontamination protocols or disposed of after one use.

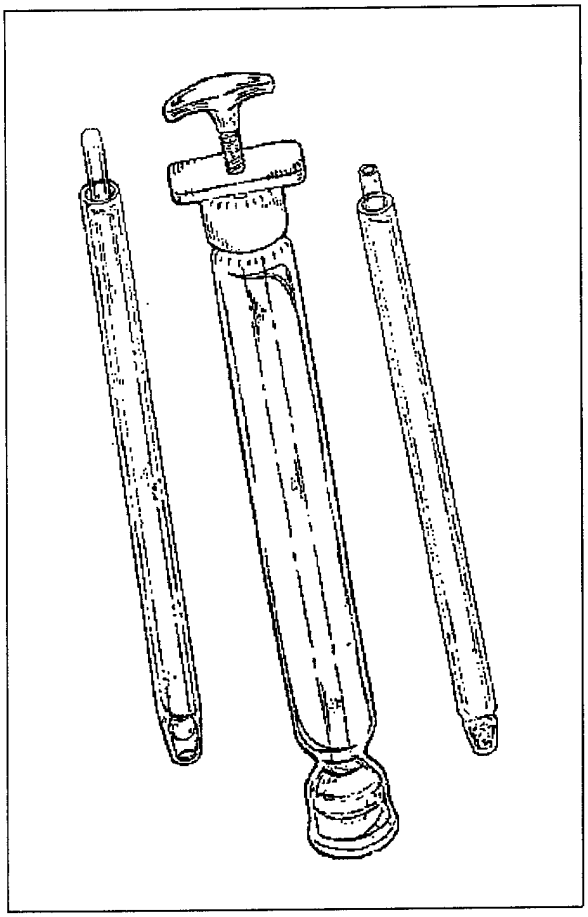


Figure D-8. Glass type Composite Liquid Waste Sampler (COLIWASA)

D.1.4.4 Scoop or trowel. Method Reference: ASTM D 5633.

D.1.4.4.1 Applicability. Stainless steel trowels can be used for sampling soil/solids drum materials and granular or powdered materials in bins or other shallow containers. The laboratory scoop, however, is a superior choice because it is usually made of materials resistant to corrosion or chemical reactions, thus lessening the probability of sample contamination.

D.1.4.4.2 Method summary and equipment. The trowel or scoop can be used to collect shallow samples in a variety of containers (see Figure C-10).

D.1.4.4.3 Sampling procedure.

- Insert scoop or trowel into material and remove sample.

- Begin sampling with the acquisition of any grab volatile organic compound (VOC) samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4, "Collection, Handling, and Storage of Solid Samples for VOC Analysis."
- If homogenization of the sample location is appropriate for the remaining analytical parameters, transfer to a stainless steel bowl for mixing. Refer to Instruction E-2 for homogenizing procedures.

- Transfer sample into an appropriate sample bottle with a stainless steel spoon or equivalent.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents, drum log sheets, and/or records in field logbooks (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

D.1.4.5 Sampling trier. Method References: ASTM D 5451 and EPA/540/P-91/008 (SOP #2017).

D.1.4.5.1 Applicability. The sampling trier is used to obtain a core sample and is preferred when the sampling medium is moist or sticky. It can be used for powdered, granular, or soil samples in relatively shallow containers; however, powdered or granular materials may provide low yield.

D.1.4.5.2 Method summary and equipment. Solid samples from open containers can be readily collected by pushing the trier into the medium and cutting the desired core sample (Figure D-9).

D.1.4.5.3 Sampling procedure.

- Insert the trier into the waste material at a 0- to 45-deg angle from horizontal. This orientation minimizes the spillage of sample from the sampler. Extraction of samples may require tilting of the containers.
- Rotate the trier once or twice to cut a core of material.
- Slowly withdraw the trier, making sure that the slot is facing upward.

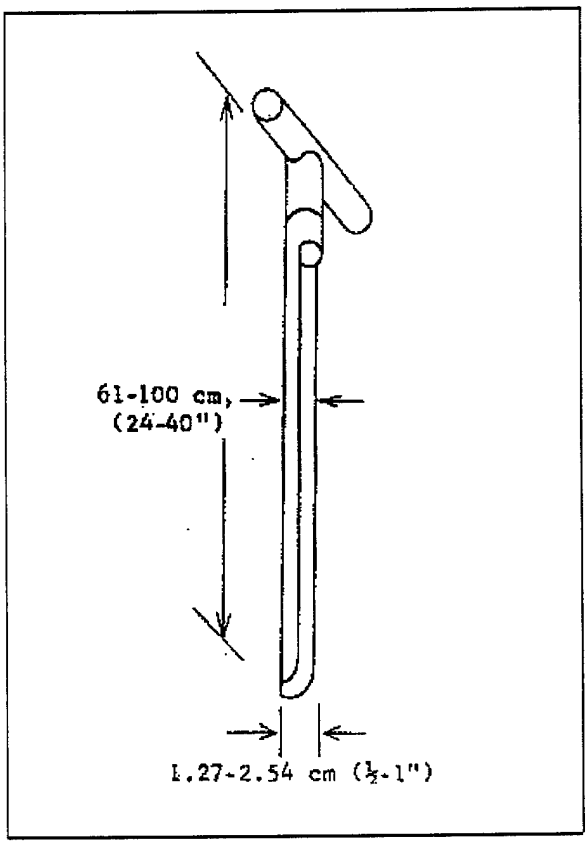


Figure D-9. Sampling trier

- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4, "Collection, Handling, and Storage of Solid Samples for VOC Analysis."
- Repeat the sampling at different points two or more times and combine the samples in the same sample container.
- If homogenization of the sample location is appropriate for the remaining analytical parameters, transfer to the stainless steel bowl for mixing. Refer to Instruction E-2 for homogenizing procedures.
- Transfer sample into an appropriate sample bottle with a stainless steel spoon or equivalent.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents, drum log sheets, and/or record in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

D.1.4.6 Grain sampler. Method Reference: EPA/540/P-91/008 (SOP #2017).

D.1.4.6.1 Applicability. The grain sampler is used to sample powdered or granular wastes or materials in bags, fiber drums, sacks, or similar containers. This sampler is most useful when the solids are no greater than 0.6 cm (0.24 in.) in diameter. It is generally used for noncohesive materials.

D.1.4.6.2 Method summary and equipment. Samples from granular and powdered materials can be easily obtained with a grain sampler (Figure D-10).

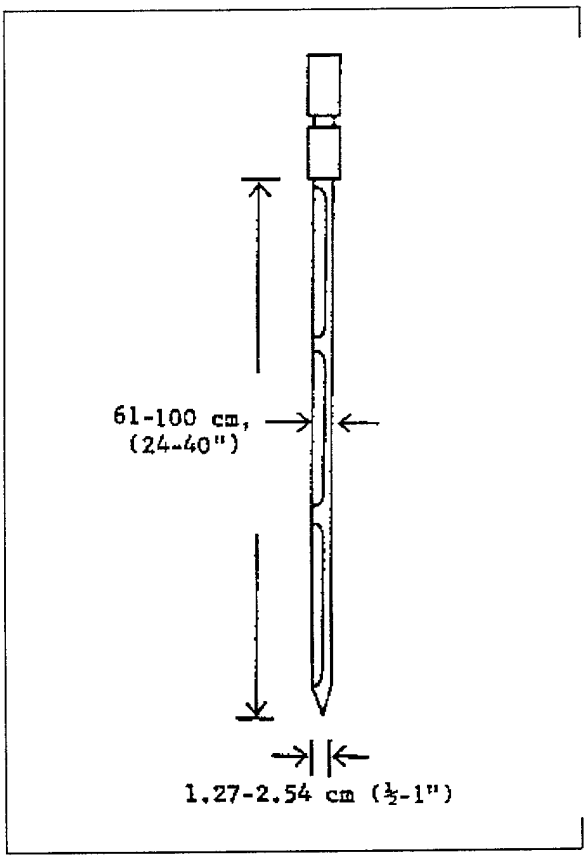


Figure D-10. Grain sampler

D.1.4.6.3 Sampling procedure.

- While the sampler is in the closed position, insert it into the granular or powdered material or waste being sampled from a point near a top edge or comet, through the center, and to a point diagonally opposite the point of entry.
- Rotate the inner tube of the sampler into the open position.
- Wiggle the sampler a few times to allow materials to enter the open slots.
- Place the sampler in the closed position and withdraw the sampler from the material being sampled.
- Place the sampler in a horizontal position with the slots facing upward.
- Rotate and slide the outer tube from the inner tube.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4, "Collection, Handling, and Storage of Solid Samples for VOC Analysis."
- Collect two or more core samples at different points and combine the samples in the same container.
- If homogenization of the sample location is appropriate for the remaining analytical parameters or if compositing of different locations is desired, transfer the sample to the stainless steel bowl for mixing. Refer to Instruction E-2 for homogenizing procedures.
- Transfer sample into an appropriate sample bottle with a stainless steel spoon or equivalent.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents, drum log sheets, and/or record in field logbooks (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

D.1.4.7 Waste pile sampler. Method Reference: ASTM D 5451.

D.1.4.7.1 Applicability. The waste pile sampler is essentially a large sampling trier and is used primarily for sampling wastes in large heaps with cross-sectional diameters greater than 1 m (3 ft).

D.1.4.7.2 Method summary and equipment. Solid samples from large containers or heaps can be collected by pushing the sampler down into the medium and then retracting the device to obtain a core sample (Figure D-11).

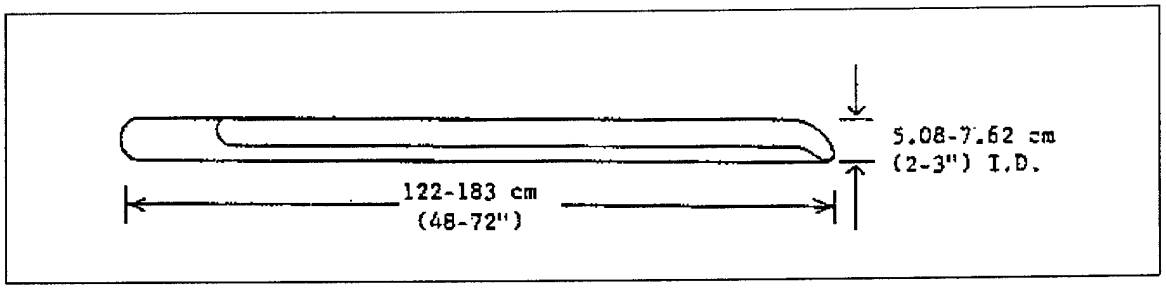


Figure D-11. Waste pile sampler

D.1.4.7.3 Sampling procedure.

- Insert the sampler into the waste material being sampled at 0 to 45 deg from horizontal.
- Rotate the sampler two or three times in order to cut a core of the material.
- Slowly withdraw the sampler, making sure that the slot is facing upward.
- Begin sampling with the acquisition of any grab VOC samples, conducting the sampling with as little disturbance as possible to the media. Refer to Instruction E-4, "Collection, Handling, and Storage of Solid Samples for VOC Analysis."
- Repeat the sampling at different sampling points two or more times and combine the samples in the same sample container.
- If homogenization of the sample location is appropriate for the remaining analytical parameters, transfer to the stainless steel bowl for mixing. Refer to Instruction E-2 for homogenizing procedures.
- Transfer the sample into an appropriate sample bottle with a stainless steel spoon or equivalent.
- Check that a PTFE liner is present in the cap. Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents and record in field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

D.1.4.8 Veihmeyer sampler. Method Reference: ASTM D 4700.

D.1.4.8.1 Applicability. The Veihmeyer sampler is used to sample deeper soils, large heaps, and containers that contain hard substances. It is designed to penetrate specific types of media without pushing the medium ahead of it, thus preventing the core from compacting in the tube.

D.1.4.8.2 Method summary and equipment. Core samples from large heaps or hard, crusty materials can be obtained with a Veihmeyer sampler (Figure D-12).

D.1.4.8.3 Sampling procedure.

- Assemble the sampler by screwing in the tip and the drive head on the sampling tube.
- Insert the tapered handle (drive guide) of the drive hammer through the drive head.
- Place the sampler in a perpendicular position on the material to be sampled.
- With one hand holding the tube, drive the sampler into the material to the desired sampling depth by pounding the drive head with the drive hammer. Do not drive the tube further than the tip of the drive guide of the hammer.

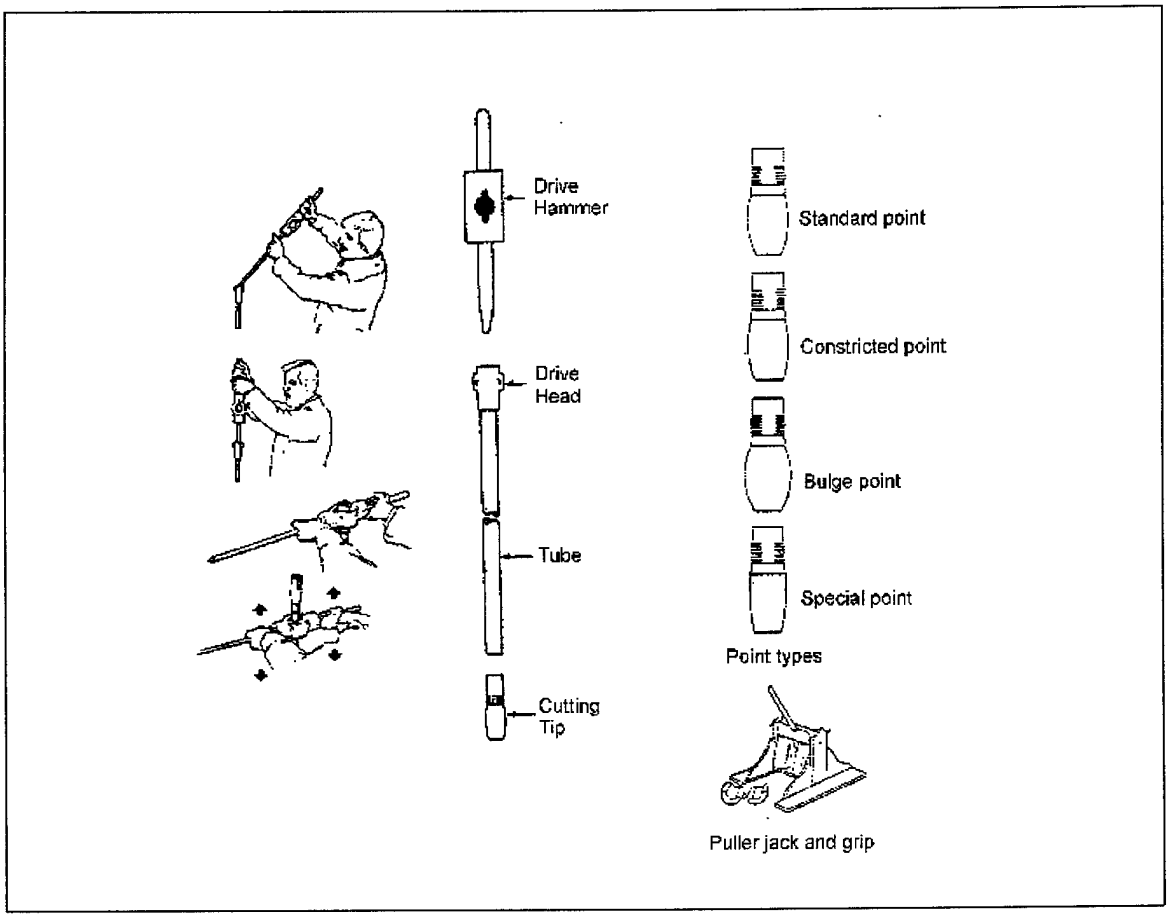


Figure D-12. Veihmeyer sampler

- Record the length of the tube that penetrated into the media.
- Remove the drive hammer and fit the keyhole-like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler.
- Rotate the sampler at least two revolutions to shear off the sample at the bottom.
- Lower the sampler handle (hammer) until it just clears the two earlike protrusions on the drive head and rotate about 90 deg.

- Withdraw the sampler from the material by pulling the handle (hammer) upward. When the sampler cannot be withdrawn by hand, as in deep sampling, use the puller jack and grip.

- Dislodge the hammer from the sampler, turn the sampler tube upside down; tap the head gently against the hammer; and carefully recover the sample from the tube. The sample should slip out easily.

- Store the core sample, preferably in a rigid, transparent, or translucent plastic tube when observation of soil layers is to be made. The use of the tube will keep the sample relatively undisturbed. In other cases, use a 1,000- or 2,000-ml (1-qt or 1/2-gal) sample container to store the sample.

- Collect additional core samples at different points.

- Secure the cap tightly.

- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

- Complete all chain-of-custody documents and record in field logbooks (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

- Decontaminate sampling equipment after use and between sampling locations.

D.1.5 Decontamination procedures. All sampling equipment must be decontaminated before its use. Much of the sampling equipment used for bulk sampling may be disposable. When applicable, sampling equipment should be cleaned as described in Instruction E-6 (Appendix E). Sampling equipment should be placed in plastic bags until immediately before use. Additional sampling devices may be needed onsite to ensure an adequate drying time.

D.1.6 Field control sample requirements. Field control samples are not routinely collected when sampling bulk materials. If appropriate, the sampling team should determine whether the data users require the acquisition of field control samples (i.e., blanks and replicate samples). A detailed discussion of field control samples is contained in Instruction G-2 (Appendix G).

D.1.7 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site set-up, sample collection, and handling as outlined previously and in Instruction F-1 of Appendix F.

D.1.8 Drum contents/waste testing. Physical and chemical HAZCAT testing should be performed on all unknown materials in order to properly characterize and segregate the contents of each drum into various waste-stream types or groups. In addition, wastes included within a hazardous waste group or within compatible groups should undergo compatibility testing before physical bulking operations. The following guidelines are provided to aid technical teams in developing site-specific physical, HAZCAT, and compatibility testing protocols.

D.1.8.1 Initial inspection record. Record any information gathered from the drum, such as the following:

- Drum size (5, 10, 18, 30, 42, 55, 85-gallon).

- Drum type (fiber, steel, stainless steel, nickel, polyurethane, polyurethane-lined, closed top, ring top, overpacked, etc.).
- Drum markings (manufacturer or chemical names, hazard symbols, or other labels).
- Drum condition (note any signs of deterioration such as corrosion, leaking, swelling/bulging).
- Field monitoring instruments readings.

D.1.8.2 Preliminary waste characterization testing. After opening, an initial sample should be withdrawn to inspect the contents. Record the sample identification number and description on the drum contents within the field logbook or drum log sheets. Recommend documenting the following waste physical descriptions:

- Number of phases: provide number and description of phases present. This determination may sometimes be difficult for very viscous liquids or resins. Lateral illumination, density, or conductivity measurement may be required to establish the actual interface in the mixture.
- Physical state: describe whether each phase is an aqueous liquid, gel, grease, oil, sludge, solid, etc. Also provide a general consistency of waste, if appropriate.
- Color: the color of each layer should be provided within the description.
- Clarity: the clarity of each layer should be described, such as clear, cloudy, translucent, or opaque.
- Homogeneity: the homogeneity of each layer should be provided within the description.
- Thickness of layer(s): the thickness of layers should be estimated from the general profile within the sampling device.
- General observations: note any additional items with respect to the appearance of each layer (i.e., dust, grains, metallic filings, fibers or friable material, pellets, crystals, fuming, etc.)

D.1.8.3 Hazardous waste characterization testing. Upon retrieving a sample, the sampler should conduct the following procedure for hazardous characterization testing as appropriate. Figure D-13 shows an example of a Hazardous Waste Characterization Testing Scheme. Each phase of a multiphase waste should be tested and classified. HAZCAT testing procedures may be purchased as commercial field kits, necessary reagents brought to a field/mobile laboratory, or samples sent to an offsite laboratory to conduct the testing.

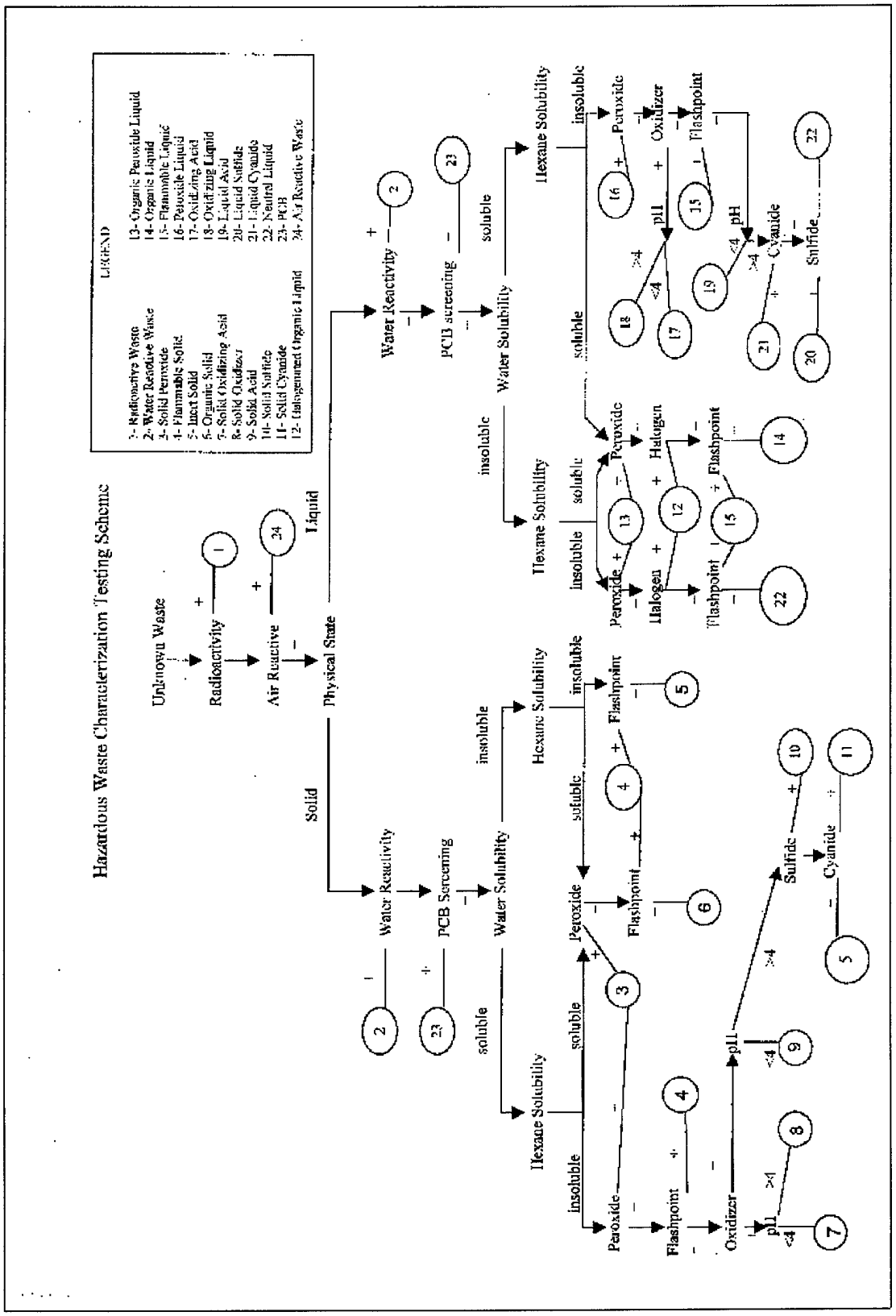


Figure D-13. Hazardous waste characterization testing scheme example

- Air reactivity
- Water reactivity
- Physical state (solid/liquid)
- Water solubility testing
- Hexane solubility testing
- pH testing
- Volatile organics headspace test with flame ionization detector/photoionization detector
- Peroxide test
- Oxidizer test
- Halogen test (Beilstein)
- Ignitability test (flashpoint)
- Cyanide test
- Sulfide test
- Polychlorinated biphenyls (PCB) screening (commercially available kits or gas chromatograph)

D.1.8.4 Waste compatibility testing. Following the physical/chemical testing, aliquots from containers with the same characteristics should be combined in a documented sequence. Visual observations (i.e., color, precipitation, gas evolution, or phase separation) and temperature measurements (to test for chemical reactions) should be made. If a reaction occurs, the compatibility sequence should be started again, omitting the sample that caused the reaction. This should be continued until the volume of waste deemed compatible is sufficient to constitute a bulk load for disposal.

D.2 Transformer Sampling

D.2.1 Scope and application. Instructions presented in this section are for collecting representative samples from transformers. Transformers can be classified into two primary types: pole-mounted and ground-mounted. Ground-mounted transformers are usually rectangular and are mounted on the ground surface or, in some cases, below ground. Pole-mounted transformers are usually round or oval and are typically mounted above ground. Instructions for sampling transformers by the following techniques are included in this section: outlet sampling method, glass thieving tube, and COLIWASA.

D.2.2 Sampling strategies. Successful investigations of hazardous waste sites are highly dependent on an effective sampling scheme. Development of a sampling scheme to characterize hazardous waste transformer contents should follow the fundamentals of scientific approach. A successful sampling scheme requires a logical design to allow an evaluation of potential contaminants.

D.2.2.1 Sampling locations. Sampling transformers is somewhat different from sampling other bulk objects. Transformers are often in secured, out-of-the-way locations, and access may present problems. Transformers may be located in underground cells.

D.2.2.2 Types of samples. Samples collected from transformers are typically discrete samples. A discrete (grab) sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected immediately and at a particular point in the sample matrix. Representativeness of such samples is defined by the nature of the materials being sampled. Composites are nondiscrete samples composed of two or more specific aliquots collected at various sampling locations and/or different points in time. Analysis of this type of sample produces an average value and can, in certain instances, be used as an alternative to analyzing a number of individual grab samples and calculating an average value. The objective of sampling transformers is to identify the potential regulatory status of the equipment and the potential presence of PCBs in the dielectric fluid contained within the transformer. Therefore, the only appropriate sample type is the discrete sample. Compositing could mask the presence of contaminants by diluting isolated concentrations of the contaminant.

D.2.2.3 Suggested samplers. Each sampling technique presents various disadvantages and advantages for its application. For example, sample disturbance, sample volume, chemical/physical reactivity between potential contaminants and sampling tool materials, and ease of use and decontamination vary from technique to technique. Discussions of the advantages and disadvantages of each sampling technique are presented in the following sections.

D.2.2.4 Sample frequency. Transformer sampling requires one sample per transformer to assess the potential hazardous nature (PCB content) of the transformer fluid.

D.2.3 Sample preservation and handling. Preservation methods and sample containers should be as prescribed in the analytical method or as identified by the analytical laboratory. Since transformer fluids are typically an oil matrix, the preservation method would be cooling. Procedures and techniques for transporting the samples to the offsite laboratory are discussed in Instruction F-2, "Packaging and Shipping Procedures," Appendix F. Improper sample handling may alter the analytical results of the sample. Samples should be transferred in the field from the sampling equipment directly into the container that has been specifically prepared for that analysis or set of compatible parameters.

D.2.3.1 Sample containers. When sampling transformers, use 125-m• wide-mouth glass jars with PTFE caps. Containers should be cleaned prior to sampling. Instruction E-6, "Decontamination Procedures," contains additional information on appropriate glassware-cleaning protocols. If precleaned bottles are used, the

cleanliness of each lot of precleaned bottles should be verified by the container supplier or in the laboratory and appropriate paperwork (i.e., certificates) be retained with other field documentation.

D.2.3.2 Sample preservation. Methods of sample preservation are relatively limited and are generally intended to retard biological action and hydrolysis and to reduce sorption effects. Preservation methods are limited to cooling.

D.2.3.3 Special precautions. Transformer sampling typically involves sampling medium- to high-level contaminants. Consequently, the following special precautions are warranted and should be taken when preparing for and performing transformer sampling:

- The transformer must be certified as "off-line" and de-energized by an electrician or other responsible person. Do not pursue any sampling activities until the transformer is disconnected.
- A clean pair of new, disposable gloves should be worn each time a different location is sampled, and gloves should be donned immediately prior to sampling.
- Sample containers for source samples or samples suspected of containing high concentrations of contaminants should be placed in separate plastic bags immediately after collecting, preserving, tagging, etc. Also segregate the samples of waste or highly contaminated samples from the ice chest used to ship environmental samples. It is also good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants should be lined with new, clean plastic bags.
- If possible, one member of the field team should take all the notes and fill out sample tags, field sheets, etc., while the other members collect all of the samples.
- Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.
- Field personnel should use sampling equipment constructed of PTFE, stainless steel, or glass that has been properly precleaned.

D.2.4 Sampling methods. Presented in the following sections are sampling instructions for the most common techniques for collecting transformer samples. Prior to sample collection, the transformer location and any specific markings or information should be recorded in the field logbook. Selection of sampling equipment is usually based on access to the transformers. If possible, obtain access to the contents of the transformer by removing the cover. A COLIWASA or glass thieving tube should be used for sampling. The outlet sampling method should be used only if other sampling methods are not possible. The toxic nature of PCBs and degree of hazard posed by their potential presence in a transformer dictate that a high level of caution should be used. As noted in Section D.2.3.3, once the power source to the transformer is disconnected, spill control measures are put in place, and the cover of the transformer, if accessible, removed with hand tools. Spill prevention control should be accomplished by using plastic sheeting on the ground and/or the floor surface of the lift, and sorbent pads.

D.2.4.1 Outlet sampling method.

D.2.4.1.1 Applicability. Sampling from the transformer outlet valve is probably the easiest method of transformer sampling. However, because the outlet valve is typically located at the base of the transformer,

sample stratification may be a problem. PCBs are generally heavier than other insulating oils and may sink to the bottom, preventing the collection of representative samples utilizing the transformer outlet.

D.2.4.1.2 Method summary and equipment. A clear, plastic (Tygon) tube is placed over the transformer outlet, and the outlet is opened allowing the oil to flow from the tube into a sample jar. It is usually advisable to have a catch bucket below the outlet to capture any spilled oil.

D.2.4.1.3 Sampling procedure.

- Install bucket or pan under the electric equipment sampling outlet to catch overflow oil.
- Obtain clear, plastic tubing (Tygon). Attach one end of the tube to the electrical equipment sampling outlet valve and place the other end of the tube in the sample container. The tubing between the transformer and the container should be as short as possible to avoid leakage potential. The tube should be of a smaller diameter than the valve ends to ensure that there is no leakage.
- Drain some oil through the sample valve cock and tubing into the overflow bucket or pan to ensure that no contaminants are present in the sampling line. Then close the sample valve cock.
- After draining some oil through the sampling line, place the tubing in the sample container.
- Open the sample valve cock on the transformer.
- Fill the sample container.
- When the sample container is completely full of oil, close the transformer valve.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents and record them in the field logbook (see Instruction F-1, "Documentation," Appendix F). Prepare sample for shipment (see Instruction F-2, Packaging and Shipping Procedures," Appendix F).
- Decontaminate sampling equipment after use and between sampling locations.

D.2.4.2 Glass thieving tube. Method Reference: ASTM D 5743.

D.2.4.2.1 Applicability. The glass thieving tube is relatively inexpensive and easy to use, but it requires removing the transformer cover.

D.2.4.2.2 Method summary and equipment. The glass thieving tube typically consists of a 6- to 16-mm inside diameter (ID) (1/4- to 5/8-in. ID) 1.2-m- (48-in.-) long glass tube. To sample, the cover of the transformer is removed, and the glass thieving tube is lowered into the oil.

D.2.4.2.3 Sampling procedure.

- Remove the cover from the transformer.
- Insert glass tubing almost to the bottom of the transformer. Approximately 0.3 m (1 ft) of tubing should extend above the drum.
- Allow the oil in the transformer to reach a constant level in the tube during the descent of the tube.
- Cap the top of the sampling tube with a tapered stopper or gloved thumb, ensuring that the liquid does not come in contact with the stopper or thumb.
- Carefully remove the capped tube from the transformer and insert the uncapped end into the sample container, being careful not to spill any oil outside the container.
- Release the stopper or thumb and allow the oil to drain into the sample container until it is approximately two-thirds full.
- Remove the tube from the sample container and dispose of the tube properly.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample tag. Be sure to label the tag carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents and record them in field logbooks (see Instruction F-1, "Documentation," Appendix F). Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).

D.2.4.3 Composite liquid waste sampler (COLIWASA). Method References: ASTM D 5495 and EPA/540/P-91/008 (SOP #2009).

D.2.4.3.1 Applicability. The COLIWASA is capable of obtaining a representative sample of multiphase containerized liquids. In comparison with the other transformer sampling methods, it is more expensive and decontamination is more difficult.

D.2.4.3.2 Method summary and equipment. The COLIWASA is designed to permit collection of representative samples from multiphase containerized liquids. COLIWASAs are commercially available and typically consist of a 1.5-m (5-ft) by 40-mm (1-1/2-in.) section of tubing with a neoprene stopper at one end attached by a rod running the length of the tube. Manipulation of the locking mechanism opens and closes the sampler by raising and lowering the neoprene stopper.

D.2.4.3.3 Sampling procedure.

- Choose the plastic (Figure D-7) or glass ball type stopper (Figure D-8) COLIWASA for the liquid waste to be sampled and assemble the sampler.
- Ensure that the sampler is functioning properly. Adjust the locking mechanism if necessary to make sure the stopper provides a tight closure.
- Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the locking block of the sampler. The open position for the

glass ball type is achieved by pulling up on the inner rod, thereby pulling the glass ball away from the tapered end of the outer tube.

- Remove the cover from the transformer.
- Slowly lower the sampler into the liquid waste. (Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same. If the level of the liquid in the sampler tube is lower than the level outside the sampler, the sampling rate is too fast and will result in a nonrepresentative sample.)
- When the sampler stopper hits the bottom of the transformer, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the closed position by turning the T-handle until it is upright and one end rests tightly on the locking block. The closed position for the glass ball type is achieved by pushing the glass ball end of the inner rod against the tapered end of the outer tube.
- Slowly withdraw the sampler from the transformer with one hand while wiping the sampler tube with a disposable cloth or rag with the other hand.
- Carefully discharge the sample into a suitable sample container until it is approximately two-thirds full by slowly opening the sampler. This is done by slowly pulling the lower end of the T-handle away from the locking block while the lower end of the sampler is positioned in a sample container. Repeat as necessary.
- Secure the cap tightly.
- Label the sample bottle with the appropriate sample label. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.
- Complete all chain-of-custody documents and record them in the field logbook (see Instruction F-1, "Documentation," Appendix F).
- Prepare samples for shipment (see Instruction F-2, "Packaging and Shipping Procedures," Appendix F).
- Decontaminate the sampling equipment after use and between sampling locations. Since the tube is small and difficult to decontaminate, it may be cost-effective to dispose of the sampler and to use new ones for additional sampling.

D.2.5 Decontamination procedures. All equipment must be decontaminated before its use. The inside surface of tubing apparatus may be considered disposable, or must be decontaminated by drawing the decontamination solution through the equipment. Other sampling equipment presented may also be considered disposable. When applicable, sampling equipment should be cleaned as described in Instruction E-6 (Appendix E). Sampling equipment should be placed in plastic bags until immediately before use. Additional sampling devices may be needed onsite to ensure an adequate drying time.

D.2.6 Field control sample requirements. Field control samples are not routinely collected when sampling transformers. If appropriate, the sampling team should determine whether the data uses require the acquisition of field control samples (i.e., blanks and replicate samples). A detailed discussion of field control samples is contained in Instruction G-2 (Appendix G).

D.2.7 Documentation requirements. Bound field logbooks should be used for the maintenance of field records. Record all aspects of the site set-up and sample collection and handling as outlined previously and in Instruction F-1 of Appendix F.

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

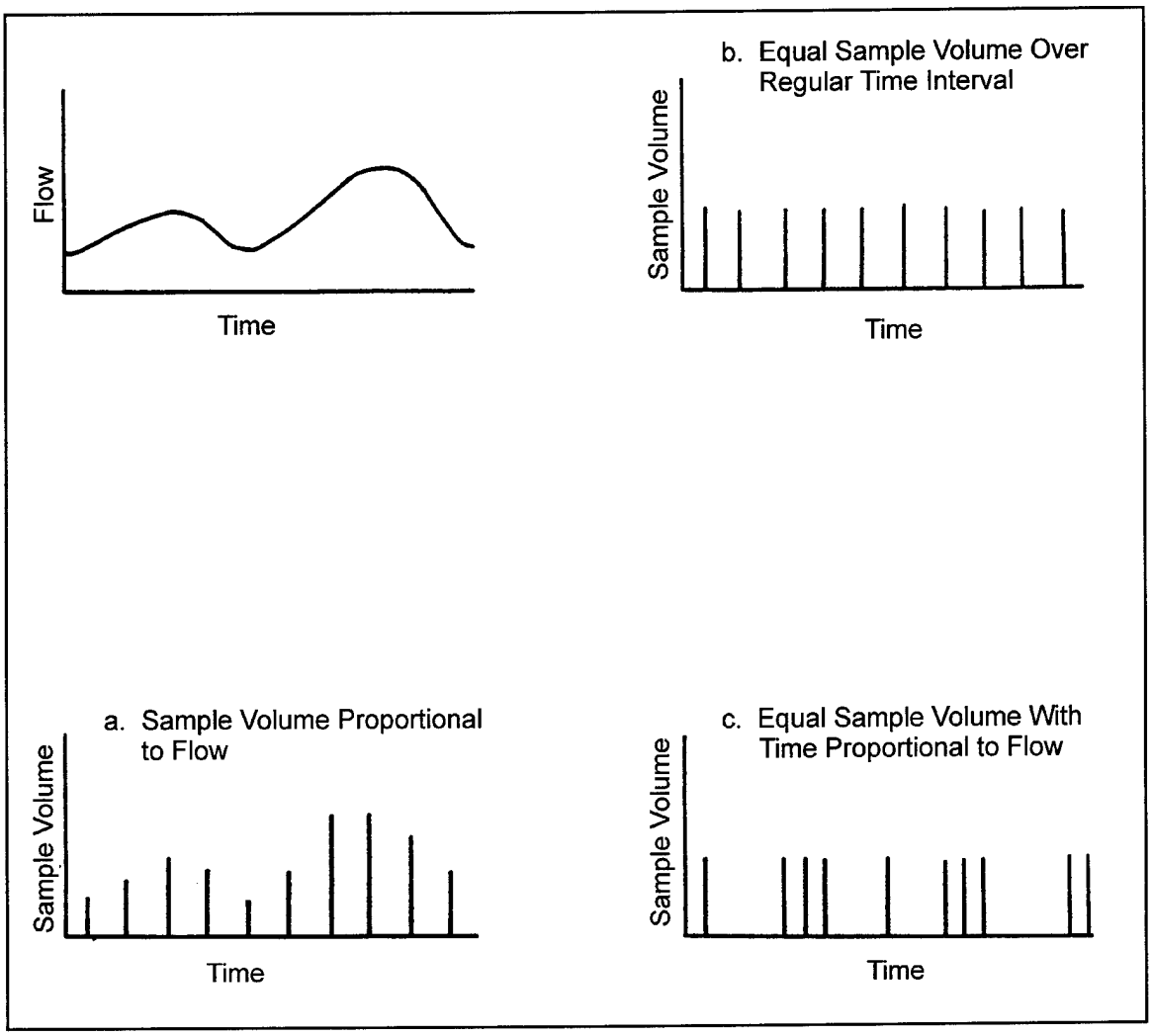


Figure E-1. Composite sampling methods

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

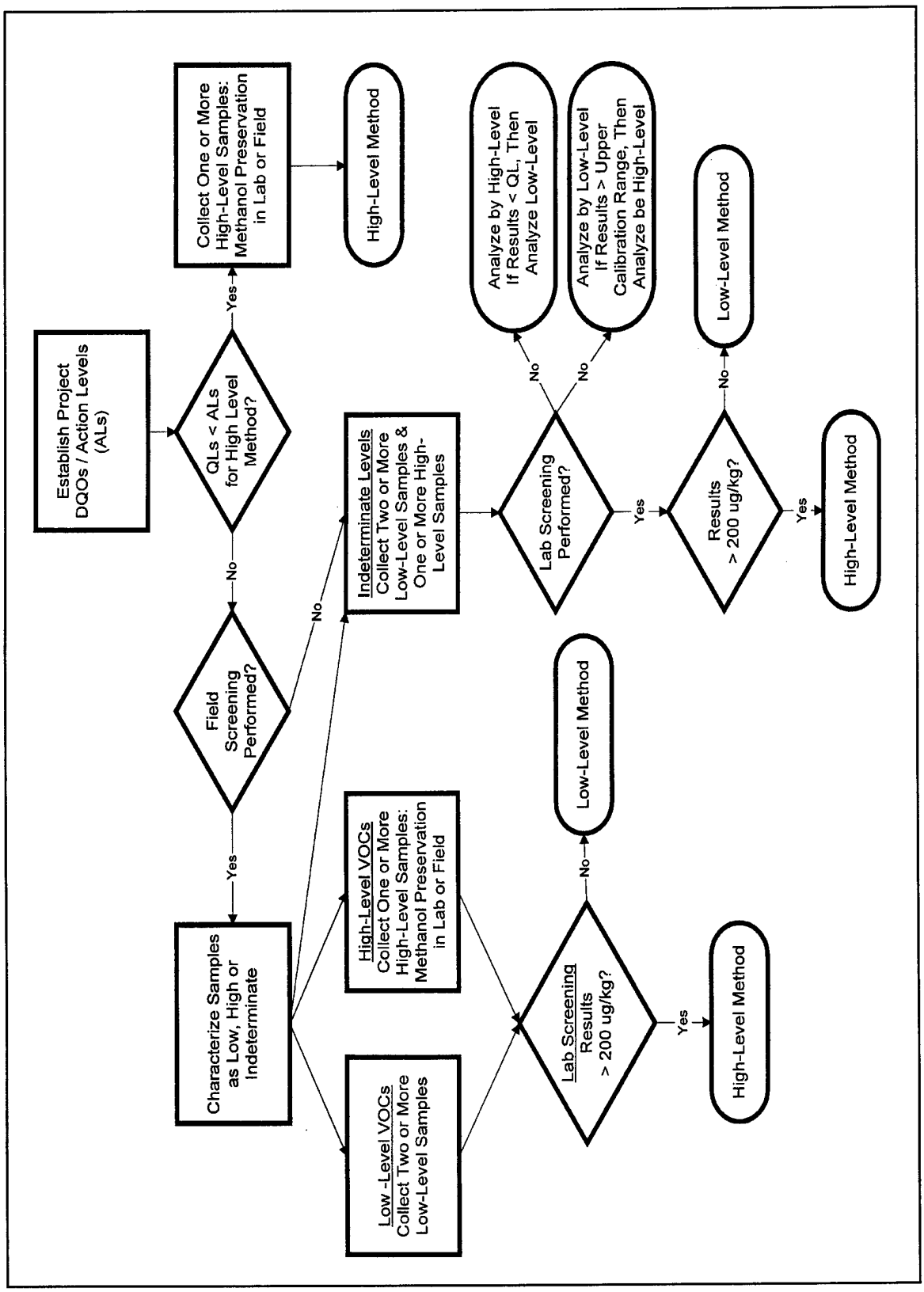


Figure E-2. VOC analysis decision tree

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:

- Project Code. An assigned contractor, project number, site name.
- Station Number. A unique identifier assigned to a sampling point by the sampling team.

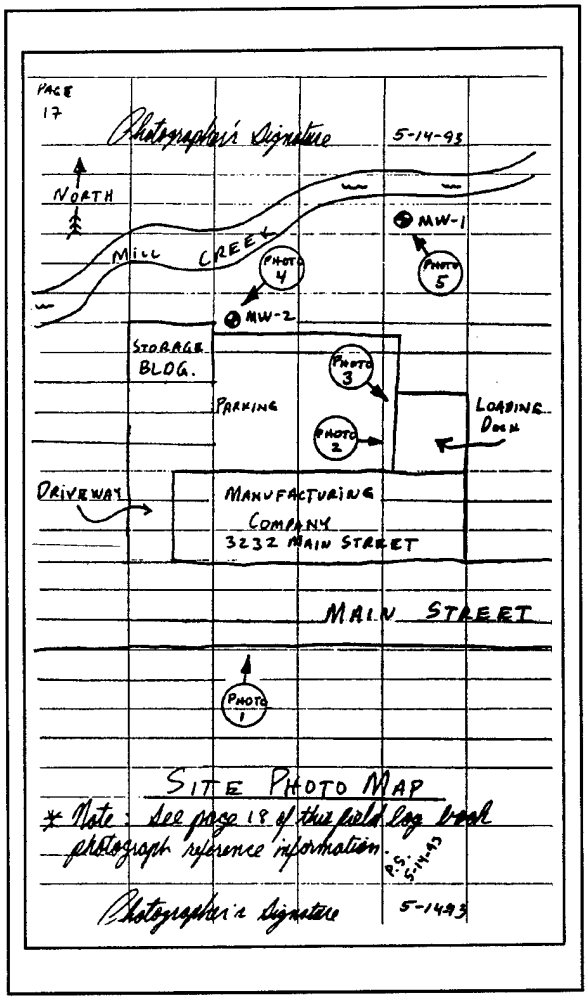


Figure F-1. Site photo map

- 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.

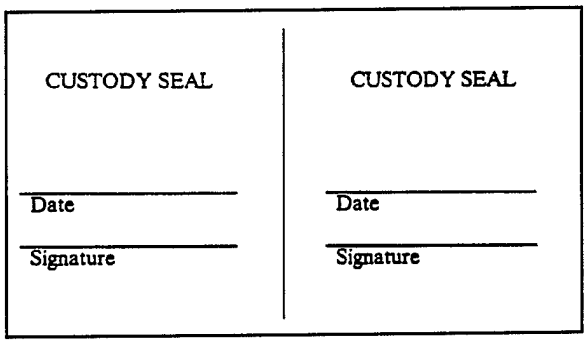


Figure F-3. Chain-of-custody seal

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.

Appendix G
Quality Assurance/Quality Control Procedures

G.1 Contractor Quality Control (CQC) Three-Phase Control Process

G.1.1 Scope and application. The contractor is required to ensure that a quality control program is in place that assures that sampling and analytical activities and the resulting chemical parameter measurement data comply with the data quality objectives (DQOs) and the requirements of the Sampling and Analysis Plan (SAP). This quality control program must be maintained throughout all field and laboratory work by means of a three-phase control process (Engineer Regulation (ER) 1180-1-6, Engineer Pamphlet (EP) 715-1-2. Corps of Engineers Guide Specification (CEGS) 01450 and 01451). The contractor quality control (CQC) process encompasses review of project activities by a contractor-assigned quality assurance (QA) officer at three distinct phases (preparatory, initial, and follow-up). This QA officer must perform these duties whether or not a Government representative is present. All CQC activities are then summarized within the contractor daily quality control reports as outlined in Instruction F-1 of Appendix F. The frequency of implementation is specified by each definable feature of work. A definable feature of work is a task that is separate and distinct from other tasks and has separate control requirements. For example, the definable features of the sample collection task include, at a minimum, each matrix (air, water, soil, containerized waste, etc.) being sampled. In addition, the quality control process shall ensure that minimum data reporting requirements are achieved and shall be implemented according to project requirements.

G.1.2 Preparatory phase. The CQC representative in conjunction with the contractor's sampling team will conduct the preparatory phase inspection prior to the beginning of any definable feature of the work. It includes a review of all work requirements, a physical examination of all required materials and equipment, an examination of work areas to ascertain completion of all preliminary work, and a demonstration of all field activities. If new sampling or technical personnel arrive onsite during the work effort, the CQC representative must repeat the preparatory phase with these personnel prior to their beginning work. All contractor personnel shall have reviewed in detail the SAP, including the Field Sampling Plan and the Quality Assurance Project Plan, prior to this inspection, and will participate in a discussion of all pertinent sections of these plans and/or specifications during the preparatory meeting.

G.1.2.1 Checklist of field equipment and other materials. The following represents a generic checklist of required onsite materials, which should be verified during a preparatory phase inspection. Also presented is a cross-reference to appropriate instructions or appendices within this engineer manual that can support a more detailed inspection. This checklist should be modified as appropriate to accommodate site conditions and be included within the SAP.

G.1.2.1.1 Project plans, and contractual documentation should be present onsite. Verify, as appropriate, that the following are the most recent/approved versions (Chapters 2 and 3):

- Contract plans and specifications
- Project plans: Work Plan, SAP, Site Safety and Health Plan, Standard Operating Procedures (SOPs)
- Summary of QA Elements and associated Measurement Quality Objectives
- Area maps for site identification and documenting sampling locations

G.1.2.1.2 Project logbooks, forms, logs, and tables should be available for documenting tasks as needed (Instruction F-1). The following are examples:

- Field logbooks/indelible ink pens
- Soil boring logs
- Monitoring well installation logs
- Drum logs
- Sample summary tables, corresponding field samples to field control samples (i.e., Tables G-1, 3-3)
- Field instrument calibration tables (Table 3-4)
- Tables for recording of any field data generated
- CQC reports
- Shipping container checklist (i.e., Figure 3-2)
- Chain-of-custody forms (i.e., Figure F-2)
- Laboratory notification checklist (i.e., Figure 3-4)
- Hazardous waste manifest forms
- Sample shipping documents (e.g., air bills)
- Communication and phone logs
- Copy of ENG Form 4025, which remedial action contractors will use to transmit analytical data

G.1.2.1.3 Reference materials should be available in hard copy or by electronic means as needed.

- Technical reference books for the identification of chemical hazards
- Material safety data sheets
- Field instrument manuals
- Reference materials and regulations for proper completion of manifests

Table G-1
Example sample table

Sample Location	Sample Depth	Sample Number (Primary Lab)	QC Sample Number (Primary Lab)	Associated Trip Blank Number (Primary Lab)	Associated Rinsate Blank (Primary Lab)	Sample Number (QA Lab)	Associated Trip Blank Number (QA Lab)	Associated Rinsate Blank (QA Lab)	EPA 8260	EPA 8270	EPA 6010	EPA 8015

Note: This table is to be completed in the file based upon the actual samples taken.

G.1.2.1.4 Field instrumentation and support equipment should be available onsite as needed (Appendix H).

- Field screening instruments
- Calibration gas or standards
- Instrument operating manual and method SOPs
- Reagents and consumables
- Computer software / data station used for data reduction or interpretation
- Established procedures or contracts for instrument repair
- Contingency arrangements or backup instrumentation and equipment, and consumables for field analysis

G.1.2.1.5 Sample collection and handling equipment needed.

- SOPs available for each sampling and sample handling protocol planned (Chapter 4)
- The following sample collection equipment (Appendices C and D): direct (sampling equipment that comes in direct contact with the sample), indirect (augers, drill rods/probes—does not come in direct contact with the sample), and support equipment (drill rig, gas cylinders, generators, etc.)
- Sample handling equipment to support the following tasks: filtration (Instruction E-1), homogenizing/compositing (Instructions E-2, E-3), and PSR, PSS (Instructions E-2)
- Personal protection equipment, gloves, duct tape, etc.
- Sample containers of the types to be used for each test or chemical analysis planned for all environmental samples and any field control (quality control (QC)) samples (Appendix B)
- Labels for sample containers (Instruction F-1)
- Sample preservatives, such as acid for metals, sodium hydroxide for cyanide, methanol or sodium bisulfate for soil volatile organic analysis (VOAs), etc. (Appendix B)
- pH test strips or meter to confirm adequacy of preservation techniques (Instructions C-1, C-2, C-3)

G.1.2.1.6 Decontamination procedures should be established with the following (Instruction E-6):

- SOPs available for each protocol employed for decontamination
- Decontamination reagents and materials including soap, solvents, rinse waters, pails, brushes, paper towels, aluminum foil, etc.

- Supplies for confirmation sampling (e.g., rinsates, wipe samples)
- Containers for storage of investigation-derived wastes, if necessary

G.1.2.1.7 Sample packaging and shipment materials should be available as noted (Instruction F-2).

- Shipping container checklist (i.e., Figure 3-2)
- Sample shipping coolers
- Chain-of-custody forms (others: analysis request forms, laboratory notification checklist, cooler receipt forms (Figure 3-3), etc.)
- Instructions for laboratory on requirements for sample subsampling procedures (Instruction E-5)
- Sample packing materials, including plastic bags, peanuts, paint cans, and/or vermiculite
- Ice packs to cool sample cooler
- Temperature blanks (Instruction G-2)
- Strapping tape
- Chain-of-custody seals
- Address labels, air bill, or shipping papers, including a completed example of the sample shipping documents used
- Laboratory information: name, address, phone number, point of contact, turnaround time for the analyses
- Communication log between field and laboratory personnel. Documentation that all laboratories have been notified that the samples will be shipped and confirmation that the laboratory will accept the samples

G.1.2.2 Checklist of demonstrated activities. The contractor will also be required to demonstrate anticipated sampling procedures during the preparatory phase inspection. The following is a generic checklist of examples that may require demonstration. This listing should be modified to reflect actual conditions anticipated at the site.

G.1.2.2.1 The CQC representative shall review all pertinent sections of the plans and specifications during the preparatory meeting in order to ensure that all field personnel are cognizant of the overall project DQOs as well as any specific sampling and analysis requirements. Likewise the sampling and analysis plan should be reviewed in detail.

G.1.2.2.2 All instruments should be calibrated during the preparatory inspection meeting using certified calibration standards, gases, etc. Personnel may provide dry run of field testing on laboratory control samples, or single-blind performance evaluation (PE) samples. Frequency and contents of data reporting requirements should be discussed.

G.1.2.2.3 The sampling team should demonstrate in detail how each type of sample will be collected, using the intended sample containers, sampling equipment, decontamination, and sample handling procedures.

G.1.2.2.4 Equipment decontamination procedures will be demonstrated in detail using the proper decontamination solutions in accordance with the SAP. If a particular area is to be designated for decontamination, this should be available and established.

G.1.2.2.5 The sample numbering system, sample labeling, and sample shipment documentation requirements should be fully discussed. Recommend a full set of sample custody forms be completed to be used as a guide during sampling activities. The laboratory addresses and phone numbers should be available and recorded on the forms. Analytical test methods and sample preservation requirements will be fully discussed and recorded on the form.

G.1.2.2.6 Laboratory turnaround times shall be established and documented in the minutes of the preparatory meeting. The CQC representative shall present a tracking system to assure that all data are received in a timely manner.

G.1.3 Initial phase checklist of activities. The initial phase inspection shall be performed when sampling is first initiated for each definable feature of work. The contractor's CQC shall oversee sampling activities and review the work for compliance with contract requirements. As a minimum, this shall include the following:

G.1.3.1 The CQC representative should oversee the sampling activities and evaluate the performance for compliance with contract requirements.

G.1.3.2 Initial instrument calibration and ongoing calibrations will be observed, verified, and documented.

G.1.3.3 Field notes will be inspected to assure that all pertinent data are recorded in accordance with the contract requirements. These notes shall include the following items as a minimum:

- Date/time of sampling
- Sampler's signature
- All field screening data (calibration, sample, QC)
- Brief description of sample(s) appearance
- Sample number(s)
- Sampling location(s), including detailed sketch
- Number and type of sampling containers prepared at each location and corresponding analytical method(s) to be used
- Identification of all split samples, blind duplicate samples, rinsate samples, etc.

G.1.3.4 Individual sample labels and chain-of-custody forms will be inspected for accuracy, completeness, and consistency.

G.1.3.5 The packaging and shipping of the samples will also be inspected by the CQC representative.

G.1.3.6 The sampling team leader should complete the table that matches up primary and QA samples at the conclusion of each day of sampling and attach a copy of the contractor's daily quality control reports.

G.1.4 Follow-up phase. The contractor is required to perform follow-up phase inspections on an as-needed basis to ensure continued compliance with contract requirements until completion of that particular feature of work. General procedures and documentation are periodically checked to ensure they are complete, accurate, and consistently executed throughout the duration of the project. Inspections shall also include a review of any field data and the daily calibration log of all instruments being used. It is especially critical that confirmation sampling be closely monitored. Confirmational sample results will serve as the basis for support for DQO attainment or site closure, and the resulting data may be evidence for decisions made by the regulators or customers that the site work has been successfully completed. Therefore, the Government requires absolute assurance that the confirmation samples are properly collected, stored, packaged, shipped, and analyzed.

G.2 Field and Laboratory Control Samples

G.2.1 Scope and application. The scope and application of this instruction is to describe standard control samples that may be included within a project data collection program to support the DQOs. The samples described include field control and/or laboratory QC samples used to assess sources of error at each stage of the sampling and analytical process. The entire sequence of sample gathering, preservation, storage, and shipment has unique errors associated with it, as do the events that occur in the analytical laboratory. To minimize or consider the impact these errors have on the resulting data, a combination of unique field and laboratory QA/QC protocols and control samples are incorporated into the project data collection program based upon project DQOs. U.S. Army Corps of Engineers (USACE) policy on QA/QC implementation is addressed in ER 1110-1-263 and EM 200-1-6.

G.2.2 Field control samples. Principal elements of the sampling and field QA/QC strategy include developing a sound sampling approach based upon the intended use of the data; using sampling methodologies that allow the collection of representative samples based upon data needs; using sampling devices that minimize the disturbance or alteration to the chemical composition of the media; employing decontamination procedures that reduce cross-contamination potential between sampling points; and using proper sample containers and preservation techniques that maximize the integrity of the samples. The applicability and appropriateness of the field sampling protocol can be verified by the inclusion of a program of scheduled field control samples, such as field replicates (duplicates, splits), field blanks (rinsate (equipment), media, bottle, and trip), background (upgradient) samples, and single- or double-blind PE samples. All field control samples shall be handled exactly as the environmental samples. With the exception of matrix spikes/matrix spike duplicates (MS/MSDs), the identity of field control samples collected should be held blind to the laboratory until the data are reported. Further discussion on the unique assignment of sample numbers is contained in Instruction F-1 (Appendix F).

G.2.2.1 Field replicates. Field replicates are samples taken in quantity at a particular location or time in order to assess error associated with sample heterogeneity, sampling methodology applicability, and sample handling techniques. These replicates may be used for various purposes depending upon the intended use of the data or eventual analysis. The different types of replicates include field duplicates/triplicates, field splits, and MS/MSDs. Field QC replicate samples are collocated or homogenized replicates used to assess the field sampling precision. If sufficient field replicate data are available (minimum of eight replicates taken), statistics may then be used to identify the general heterogeneity of the media population being assessed. Similarly, field QA split samples are collocated or homogenized replicates of field samples, except that these are sent to a referee (QA) laboratory for analysis. These field split samples have been used by USACE for early detection of problems with contractor's field sampling, documentation, packaging, and/or shipping errors. Finally, the MS/MSD are collocated or homogenized replicates which are clearly identified as associated with its primary environmental sample. The MS/MSD is used to verify the applicability and effectiveness of the analytical procedures in the project matrix. In addition, this referee laboratory analysis offers a source of data which may provide special attention to the achievement of lower detection limits, allow the performance of supplementary cleanup procedures to avoid matrix interferences, or may help identify an analyte as a laboratory contaminant. These split samples are integral to the USACE Contractor's Quality Assurance Report. It should be noted that the techniques and methodology used for field replicate sample acquisition differ (collocated or homogenized), depending upon the media being sampled and the requirements of subsequent analysis. Refer to Instructions E-2, E-3, and E-4 (Appendix E) for further information on sample handling practices such as homogenization, compositing, and collocated sampling techniques.

G.2.2.1.1 Collocated (grab) replicates. Aqueous media and samples that require grab techniques (e.g., VOA) are field replicates obtained from multiple grab samples, collected separately, and placed directly into sample containers. Theoretically, each grab sample equally represents the medium at a given time and location.

G.2.2.1.2 Homogenized field replicates. Field replicates of solid matrices whose subsequent analysis allows homogenization of the media are obtained from one location in sufficient volume to fill all sample containers. The medium is homogenized and divided into equal quadrants, and equal aliquots from each quadrant are used to fill the sample containers. Refer to Instruction E-2 in Appendix E for details on this sampling technique.

G.2.2.2 Field blanks. Whenever the possibility exists for contributing extraneous material into the sample collection, shipment, or analysis, a blank sample should be used to assess the magnitude of this contribution. Blank samples associated with the field sampling effort include rinsate (equipment), media, bottle, trip, and temperature blanks.

G.2.2.2.1 Rinsate (equipment) blanks. Rinsate blanks are samples of analyte-free (deionized) water that are rinsed over decontaminated sampling equipment, collected, and submitted for analysis. These samples are used to assess cross-contamination from the sampling equipment, in addition to incidental contamination, the sample container, and/or preservatives.

G.2.2.2.2 Media blanks. Media blanks are samples collected following the same sample methodology as the environmental samples without exposure to the contaminant under investigation. Examples include samples of solvent (sodium sulfate or methanol) blanks for solid VOA samples; the filter and solvent media taken in conjunction with wipe samples; filter for vacuum samples; probe blanks for soil gas survey systems, etc. Analysis of these samples allows an evaluation of the contribution due to the media used in sample acquisition.

G.2.2.2.3 Bottle blanks. Bottle blanks are not commonly used on USACE projects; however, they may be required based upon customer or regulatory preference. Bottle blanks are analyte-free (deionized) water for aqueous samples (purified air for canister blanks) that is transferred to a clean sample container in the field and submitted for analysis. A purified solid such as a certified clean sand may also be used. These samples are used to assess the potential incidental contamination due to the field operations (exposure to air) and/or contamination due to the sample container or preservatives, if applicable.

G.2.2.2.4 Trip blanks. Trip blanks are samples of organic-free (deionized) water that are prepared in the laboratory and shipped onsite with the other sample containers. They are then returned to the laboratory unopened in each shipping container that contains aqueous VOA samples and analyzed. Trip blanks are generally prepared with water which meets American Society for Testing and Materials Standard D 1193 requirements and containerized with no headspace. Trip blanks are used to evaluate the potential cross-contamination that may occur during shipment of aqueous samples.

G.2.2.2.5 Temperature blanks. A temperature blank is a container (e.g., 40 mL) of water packaged along with field samples in the shipping cooler that will represent the temperature of the incoming cooler upon receipt at the laboratory. Use of these samples within a shipping container enables the receiving laboratory to assess the temperature of the shipment without disturbing any project field samples.

G.2.2.3 Background (upgradient) samples. Background, upgradient, or upwind samples are samples from a medium similar to that under investigation, but outside the presumed area of contamination. The

sample locations or time should be near that of the field samples but will vary depending upon media and site conditions. These samples are taken to measure the concentration of analytes considered naturally occurring, due to another contaminant source, or due to an interference present within the media itself. Background samples of each unique matrix should be acquired to evaluate the presence of analytes within the field samples. Background samples are especially recommended for complex matrices due to the interferences that may occur during analysis.

G.2.2.4 Single- or double-blind performance evaluation (PE) samples.

G.2.2.4.1 One of the ways of testing a laboratory's/analyst's proficiency in identifying and quantifying analytes of interest is the use of single- or double-blind PE samples. The composition of PE samples is known to the originator, but not the analyst. In a single-blind PE sample, both the originator and the analyst know that the sample is a PE sample. The USACE uses single-blind PE samples as part of the process to validate laboratories. In a double-blind PE sample, the sample is containerized, labeled, and submitted as an environmental sample. The analyst does not know that the sample is a PE sample. Ideally, the PE sample will be indistinguishable from the other project samples. The use of double-blind PE samples is considered a more effective way of detecting problems, since the laboratory would not be aware that it was being evaluated. Currently, commercially available PE samples may be acquired for a complete range of organic and inorganic analytes in both water and soil.

G.2.2.4.2 However, it may be difficult to disguise a standard reference sample as a project sample, and the concentration of the target analytes within the standard reference sample may not be appropriate or useful to the project. For this reason, one may consider the preparation of matrix-specific double-blind PE sample. These PE samples involve the utilization of a sample replicate to which a known quantity of analyte is added and sent blind to the laboratory. It is recommended to spike at levels that pertain to concentrations of concern or action levels associated with that compound or parameter. This type of sample is used to evaluate the laboratory's accuracy by comparing the recovery of analyte reported to the amount added. However, due to the difficulty in achieving thorough and even distribution of the spiked material within the field sample, as well as the potential for field exposure and cross-contamination of other samples due to the presence and handling of the contaminant source in the field, it is not recommended to perform these procedures in the field. Suggest the use of the USACE government laboratory to prepare these types of PE samples from the QA splits received. In addition, the USACE government laboratory can retain a portion of the material to conduct further referee analysis. PE sample data are evaluated for correct compound identification, accurate results quantitation, and assessment of any laboratory contamination. PE samples are recommended for sites that have the potential for a majority of nondetects, for sites where the contaminants of concern have already been identified, or for contracting (laboratory) firms suspected of inaccurately reporting chemical results.

G.2.3 Laboratory QA/QC procedures. Laboratory QA/QC procedures are implemented in order to prevent, detect, and correct errors in the analytical process. In order to ensure that quality data are continuously produced during all analyses, and to allow eventual compliance review, systematic checks are performed to show that test results remain reproducible and that the analytical method is actually measuring the quantity of target analytes in each sample without unacceptable bias. The reliability and credibility of analytical laboratory results are typically corroborated by the inclusion of a program of scheduled analyses of replicates, standards, reference solutions, surrogates, and/or spiked samples. It should be emphasized here that additional volumes and/or samples are required when matrix spike/matrix spike duplicate analysis is required for the project in order to assess the appropriateness and accuracy of the laboratory's analytical method with regard to the matrix under investigation. Refer to Appendix I for details on the scheduled QC procedures required for individual analyses.

Appendix H
Field Analytical Technologies

H.1 Scope of Application/Background

H.1.1 Field analytical technologies are rapidly evolving. Growth is apparent in both the number and types of techniques available, as well as in improvements in selectivity and sensitivity. These and other advances have increased the viability and application of field analytical technologies to the support of Hazardous, Toxic, and Radioactive Waste (HTRW) project execution. However, the use of field analytical technologies is too often overlooked or disregarded due to a general misconception by regulators, project personnel, and other stakeholders regarding the quality or comparability of the resulting data, and the perceived difficulty in the planning and execution of these techniques. Additionally, the project planning team members may be unfamiliar with the types of field analytical technologies and services available. Other limitations include a lack of reliable performance data for a particular environmental matrices, unknown performance under adverse field conditions, and incomplete or unreliable cost data to support an evaluation of the field analysis. This appendix will identify project-specific information needed to evaluate field analytical technologies application (Section H.2); provide general guidance for selecting appropriate technologies (Sections H.3 and H.4); provide general guidance for implementation and oversight of field analytical technologies (Sections H.5 and H.6); and identify sources for gathering specific information on field analytical technologies available and generating case studies(Section H.7).

H.1.2 Field analytical technologies are used to monitor unstable or volatile parameters (e.g., pH, redox potential, dissolved oxygen, temperature, etc.); provide health and safety information on highly contaminated areas that should be avoided or require special considerations; perform general site reconnaissance to identify hot spots or areas requiring further investigation; provide rapid or real-time data to focus the selection of samples for definitive or confirmatory analyses, or to optimize sample locations chosen for a more efficient sampling/analytical strategy; monitor remedial actions/treated waste streams to provide a timely assessment of its effectiveness; or provide an increased sample density onsite for statistical treatment, or to more thoroughly define nature and extent of contamination in an area/matrix, etc. Many times, field analytical technologies are used to support expedited site characterizations or adaptive sampling and analysis plans execution. In this way the field measurements are used in conjunction with an established decision logic to identify appropriate contingencies or actions in the field. In addition, the advent and promoted use of performance-based measurement systems and performance-based methods encourage the use of field analytical technologies as the primary analytical tool supporting project decisions, refining the project conceptual site model, etc. Field analytical technologies are easily integrated into a more cost-effective data collection option for site characterization and environmental restoration monitoring.

H.1.3 Field analytical technologies are routinely categorized as either field screening or field analyses. The specific application of either term depends on the amount of quantifiable error, level of method quality control (QC), and selectivity of the technology in question. Distinction between the terms may also be dependent on the efficiency of the preparatory procedures used to isolate analyte(s) of interest. Application of both types of field analytical technologies has utility in the U.S. Army Corps of Engineers HTRW program and will depend on the specific data need. Instrumentation used to support the generation of field screening or field analyses data may be grouped in one of the following three categories:

- Hand-held or portable: no external power source required/able to be used directly or with limited setup/typically provides qualitative or semiquantitative data

- Transportable or field portable: external power source required/able to be used with nominal facility (i.e.,van)/typically provides semiquantitative to quantitative data

- Mobile laboratory: external power source required/must be used within controlled environment and/or facility/can provide quantitative (definitive) data similar to a fixed laboratory, depending on the specific analytical *(preparatory/determinative)* methods employed

H.1.4 Regardless of whether field screening or field analyses techniques are employed, a certain percentage of the data may have to be confirmed by definitive data. The definitive data may be generated onsite at a mobile lab or at an offsite laboratory. The appropriate percentage of confirmatory analyses should be determined based on the quality of the field data generated and its intended use.

H.2 Project Data Needs and Information

H.2.1 EM 200-1-2 provides guidance on a systematic planning process to determine overall site goals, project objectives, and project data needs that are used to define the data collection program. It endorses the generation of an appropriate type and quality of data based on its intended purpose. If done properly, the project planning process may determine that application of a field analytical technology is as good, or better than fixed laboratory analyses. In order to benefit from field analytical technologies, project team members must understand the flexibility and advantages that onsite data provide in tailoring a sampling and analysis program during execution. Additionally, the technical planning team must know where to obtain information on the types of field analytical technologies available. To determine the viability of field analytical technologies application, the technical planning team should utilize the guidance established in EM 200-1-2, gleaning the following project information, and evaluate its potential utility. After an assessment of project information, the technical planning team can decide whether application of field analytical technologies is feasible and may benefit the project.

H.2.2 During EM 200-1-2 Phase One, information is gathered on prior site use, site-related contaminants, and their expected range of concentrations. Note any site constraints or features that may impact the field technologies performance. For instance, knowledge of the site location, temporal/seasonal conditions, and general site topography may define whether site constraints exist due to isolation of the site, short working seasons, or if the terrain imposes additional demands or hazards. Specifics on depth to ground water, soil type, soil moisture content, or soil organic content can provide invaluable information when evaluating applicability of a particular field analytical technique or when comparing multiple techniques.

H.2.3 During EM 200-1-2 Phase Two, project personnel articulate the appropriate project data needs and their intended purpose. Most importantly, the data user must define any applicable action or decision levels and any uncertainty, performance, or acceptance limits associated with the use of this decision level. This information is used during Phase Three by the sampling and analysis data implementors to determine the project contaminants of concern or chemical parameters to be measured. Key issues to focus on, include estimating the number of samples needed to support the data use and defining those contaminants to be monitored. The number of samples anticipated should be sufficient so that the offsite analytical costs are comparable to or greater than the costs of using a field analytical technology. For depending on the capital investment (rental or purchase of instrumentation), facility requirements, consumables, and labor costs of onsite personnel, a minimum number (e.g., 50 to 200) of samples may be necessary to support the use of the field analytical technology. When determining the contaminants to monitor, consideration should be given to the most mobile contaminants, such as those most likely to reach ground water, or are the best indicators of migration potential. Other criteria include contaminants that pose the greatest risk or may directly impact remedy selection. Evaluation of the physical and chemical characteristics (e.g., water solubility,

octanol/water coefficient (Kow), etc.) of the contaminants help to assess their fate and transport potential. Finally, if there are a number of contaminants present that may be monitored, selection should be based on the analyte that is most reliably detected by the available field analytical technologies. The above information is used to determine the project indicator compounds, and where, and from what type of media to collect the samples.

H.2.4 Preliminary project measurement quality objectives (MQOs) should be formulated to define requirements with which to evaluate applicability of field analytical technologies available. Topics include defining selectivity requirements. For instance, whether an individual target analyte(s) or a chemical compound class require monitoring. Define sensitivity (detection limit) requirements as clearly as possible, for this is typically a critical parameter depending on data use and contaminant concentrations found onsite. Any uncertainty requirements established by the data user support the specification of preliminary MQOs for precision and bias of the field analytical technologies, and to estimate the percentage of confirmatory/definitive data necessary.

H.3 Identification of Applicable Field Analytical Technologies

H.3.1 Using the sources outlined in Section H.7, knowledge of type of media to be sampled, and contaminants to be measured, identify potential field analytical technologies to use. It is important to involve technical personnel familiar with the basis of measurement for the field analytical technology (i.e., what chemical and/or physical property of the contaminant is being measured) and the factors controlling performance, to optimize performance to field matrices and understand data comparability issues. The field analytical technology may be based on existing laboratory instrumentation, modifications (e.g., microinstrumentation) to existing instrumentation, or a new technology. Use of unproven, emerging technologies may require the use of more extensive pilot studies, increased QC measures, and/or interim milestones at project initiation, to verify the viability of the field method and reinforce confidence in its data. Identify technologies possessing capabilities that, at a minimum, meet the stated project MQOs for selectivity, sensitivity, bias and precision identified from Section H.2.4. Care should be taken when interpreting vendor claims for selectivity, sensitivity, bias, and precision, for these may be based on a clean or well-homogenized matrix. Suggest using the vendor information for estimating purposes only. If any of the MQOs or performance objectives are critical to decision-making, verification of these parameters using project-specific matrix(ces) is encouraged. Typically, vendors will perform preliminary studies to verify sensitivity, establish appropriate calibration standards, or verify precision and bias at a project action or decision level with the use of project-specific matrix(ces) at nominal cost. Next gather information identified in Sections H.3.2 - H.3.4 for applicable field analytical technologies to evaluate them for potential selection or exclusion.

H.3.2 Review performance capabilities of applicable field analytical technologies. Identify media applicable to the field analytical technology or method, the calibration range, and false positive (FP) / false negative (FN) rates. If information is available, verify the spike concentrations at which the FP and FN studies were performed, assessing their relevance to project action or decision levels. The FP/FN values claimed should also be interpreted as approximate values, and project-specific FP/FN values should be determined in conjunction with execution as outlined in Section H.5.

H.3.3 Review operational characteristics of applicable field analytical technologies. These include items such as sample analysis time (estimated samples/day), capital investment necessary, consumables costs per test/number of tests, estimated labor costs, facility needs and consumable storage requirements, operator experience requirements, any training courses or certification requirements, and the portability and reliability (durability, ruggedness) of instrumentation.

H.3.4 Review limitations and interferences of applicable field analytical technologies. This includes unsuitable physical conditions and any known chemical interferences or cross reactivities.

H.4 Field Analytical Technologies Selection

Once a listing of applicable field analytical technologies are defined, the strengths and weaknesses of each are compared to support selection of the technology to employ. Review each field analytical technologies performance capabilities, operational characteristics, and limitations as defined in Sections H.3.2, H.3.3, and H.3.4 against site features, project constraints, and any project DQOs/MQOs to identify those to retain or eliminate. Additional issues to address include the suitability of the technology to site physical conditions, the commercial availability (rental versus purchase) of the equipment, the maturity of the technology (new/emerging, accepted/proven), and whether it has been applied to a variety of site conditions. Final choice of the field analytical technology to employ (all other features being equal/similar) will be defined by differences in cost. Tradeoffs for performance, quality, and cost must be reconciled among data users. Cost comparisons become more involved when complex field analyses (e.g., mobile laboratory) are being implemented. In these cases, suggest cost comparisons estimate total costs, including capital investment, facility, consumables, and labor costs compared with the cost of the remobilization of another (phased) project execution.

H.5 Field Analytical Technologies Implementation

H.5.1 Implementation of any field analytical technology requires effective upfront project planning. Decisions must be made about the extent to which to apply the following procedures. While many of the items should be considered mandatory. Others will depend on project constraints, the critical nature of the field data's use, the maturity of the field analytical technique, and the similarity or correlation between the field technique and the definitive/confirmatory analysis whether to implement them.

H.5.2 Define the appropriate field screening/analytical methods to be used. If applicable, this shall encompass both the preparatory and determinative procedures and any modifications employed. Standard operating procedures (SOPs) must be generated for review/approval by appropriate project personnel and stakeholders in conjunction with the SAP. Refer to Section 4.4.2 of Chapter 4 for potential input into the format and contents of the field analytical SOPs. While the majority of SOP sections apply to both field screening and field analytical methods, the level of rigor and mandatory frequency for these subjects (i.e., calibration, quality assurance (QA)/QC procedures, corrective actions) may be less than those prescribed within the associated definitive data methodologies. These issues should be considered negotiable and should be set at a level that supports the intended use of the data and minimizes the risk of making a wrong decision. Any project-specific action levels (concentrations) or critical decision levels should be established at midcalibration range, if possible. In addition, consider periodic verification of this level to evaluate recovery ranges observed.

H.5.3 Determine the field analytical technology's application to and performance within a project-specific matrix by implementing any or all of the following procedures. If working directly with a manufacturer or vendor, submission of project samples to the vendor may be an option to calibrate field instrumentation (i.e, X-ray fluorescence), evaluate sensitivity, bias, or precision ranges. Pilot studies are another mechanism to allow the verification of a field analytical technology to project matrices with less risk or cost than full project mobilization. If neither option is utilized, recommend, at a minimum, that an interim milestone be established to review data generated and determine the viability of the technique prior to full-scale project execution. During these interim measures, suggest implementation of the following. Determine matrix-specific detection and quantitation limits to assess sensitivity of technique. Refer to section I.3.3.7

of Appendix I for procedures to evaluate sensitivity. Verify that the sensitivity achieved is at least one-half of any project action or decision levels. If this is not attainable, suggest verifying this level with a low-level check or other sample as noted in Section H.5.2. When abbreviated or modified preparatory procedures are conducted, suggest a study be performed to verify extraction efficiency at project initiation to optimize extraction times to the contaminant and matrix. Refer to Jenkins et al. (1996b) for additional information on this subject. Employ a high percentage (75-100 percent) of redundant definitive analyses initially to establish a correlation between the field analytical and definitive measurement techniques. This preliminary correlation (or conversion factor) between field and definitive results is used to ensure usability of the field analytical technology and its data. Define the minimum amount of data to establish this correlation. If there is a significant difference between the field analytical and definitive data, apply a conversion factor to the project action or decision levels to establish an appropriate field decision level.

H.5.4 Define appropriate QA elements to apply for proper chemical data quality management on the project. Refer to EM 200-1-6 for additional information on QA elements that may be employed. For instance, mobile laboratories may be subject to validation procedures or onsite inspection; single- or double-blind PE samples may be submitted to verify accurate determination of appropriate target analytes; field QC samples may be taken (blanks, replicates, matrix spikes, etc.) to monitor sampling, sample handling practices, and field analytical procedures. As noted in Section H.5.1, the frequency of implementation should be based on the intended use of the data.

H.5.5 Define the percentage of redundant, confirmatory/definitive analyses to employ throughout the remainder of the project to evaluate the initial/current correlation is accurate and representative of the data sets. Definitive analyses may be performed onsite or offsite. Suggest varying the samples submitted for definitive analyses between collocated grab samples, homogenized replicate samples, or portions of the original sample/extract/digestate to assess various sources of sampling and analytical error. Suggest the samples sent for definitive analysis also encompass a variety of concentrations, as determined by the field results. Percentages may be applied as noted in Table H-1 or revised based on project size or DQOs. Alternatively, application may be based on whether the field results are above/below a project action level alone. Suggest a minimum of three above and three below the action level (or nondetect) be sent for redundant definitive analysis.

Table H-1
Recommended Percentage of Redundant Analyses for Confirmation Sample Analyses

Field Analytical Results (FAR) Relationship to Project Action Levels (AL) / Detection Limit (DL)	Recommended Percentage of Redundant Analyses
DL < AL < FAR	5 - 10% (allows FP assessment)
DL < FAR < AL	10 - 20%
FAR < DL < AL	10% (allows FN assessment)

H.5.6 Define appropriate field data review requirements, data reporting requirements, records archival and retention, and client notification requirements.

H.5.7 Employ data verification techniques to assess the comparability of the field and definitive data, and the useability of the field analytical data to support its intended use or other purposes. Use regression analysis or other statistical technique to compare the field analytical data to the definitive data, especially around the project action or decision levels. Results should be reviewed in light of the following

considerations: sample heterogeneity, differences in protocols for the sample preparation or analysis. Determine the FP and FN rates for the field analytical technology (as compared to definitive results) and any impact this may have on the data.

H.6 Field Analytical Technologies Oversight

H.6.1 Conduct project oversight as described in Instruction G-1 of Appendix G. This should include a preliminary site visit and inspection during the pilot study or at the inception of the project to verify the following, at a minimum:

- All personnel have clear understanding of the contingency/corrective actions/notifications scenarios which apply based on field results, and its impact on field decision making.
- Sample handling procedures and field instrumentation use are in accordance with manufacturer's guidelines and approved SOPs.
- Field data reporting frequency and content requirements are understood and in place.
- Samples sent for definitive analysis are in accordance with logic prescribed in the Sampling Analysis Plan.
- QC results are compliant with project MQOs.
- Field data review is complete, accurate, and documented.

Additional field inspections should be conducted as needed to monitor ongoing field operations and project progress.

H.6.2 The project report should be generated to compile and discuss the field analytical results and their usability to support project decisions. Suggest this information be presented to the regulators, community, and customer to enhance communication and understanding of site data. Include data comparisons performed in light of the sample heterogeneity and differences in protocols noted in H.5.2. When the correlation coefficient (r) > 0.90, the field analytical data may be considered definitive, and may be used to support compliance, no-further-action alternative, and risk assessments. Lesser correlations may still provide quantitative or qualitative support for data needs, depending on the use of the data and consistency of the correlation. Finally, suggest that the U.S. Environmental Protection Agency (USEPA) guidance identified in Section H.7.4 be used to develop a project case study for the continued education and support of field analytical technologies and their use.

H.7 Information Sources for Field Analytical Technologies

H.7.1 Field analytical technologies encompass a wide range of technologies and instrument types. In general, the majority of techniques may be divided into the following categories: geophysical, inorganic, organic, unexploded ordnance/explosives, radiochemical, and health and safety. However, each source should be reviewed to identify the categories that may be queried. Due to the diverse nature and vast number of field analytical technologies available for detecting environmental contamination, guidance on key sources for retrieval of information is provided.

H.7.2 The following websites provide sources of information for the types of field analytical technologies available for a particular contaminant and media, and/or information on vendor sources for commercial availability of the field instruments or analytical techniques.

- **FRTR** (**F**ederal **R**emediation **T**echnologies **R**oundtable)
  http://www.frtr.gov/
  This website maintains a Sampling and Analysis Matrix that provides comparative screening information for several sampling and analytical technologies. The matrix is intended to inform project personnel about the variety of technologies that are commercially available, providing a general comparison among them. Additional information is provided for each technology, including a general description, identifying applicable media, analytes/chemical parameter, selectivity, relative level of quantitation, detection limits, turnaround time, limitations, susceptibility to interference, status of technology (i.e., commercial availability), any certification/verification the technology maintains, and relative costs. The equivalent publication released at the initiation of the website is EPA/542/B-98/002, Field Sampling and Analysis Technologies Matrix and Reference Guide, March 1998.

- **FATE** (**F**ield **A**nalytic **T**echnologies **E**ncyclopedia)
  http://www.ttclients.com/encyclopedia/
  This website is intended to provide information about technologies that can be used in the field to characterize contaminated soil and ground water, monitor the progress of remedial efforts, and in some cases, and for confirmation sampling and analysis for site close out.

- EPA **REACH IT** (**RE**mediation **A**nd **Ch**aracterization **I**nnovative **T**echnologies)
  http://www.epareachit.com/
  This website replaces the VISITT, VendorFACTS, and ITT Databases. It provides information on the site characterization and monitoring options available. Information is included to identify the capabilities of vendor-specific instrumentation and technologies. Information is included on the technology description, applicability to various media, performance capabilities, and cost.

- DOE **PAM** (**P**referred **A**lternatives **M**atrix)
  http://www.em.doe.gov/define/
  This website provides a matrix to identify proven, available technologies and rank them on the basis of performance, risk of technology failure, and cost. The PAMs provide a tool for field personnel to focus remedy selection and expedite preferred alternatives implementation to allow preselection of effective, low- cost alternatives for monitoring of site contaminants.

- DOE **CMST-CP** (**C**haracterization, **M**onitoring & **S**ensor Technology-**C**ross Cutting **P**rogram) Vendor Database
  http://www.cmst.org/vendor/
  This website provides information for the chemical and physical property measurements of environmental samples. The CMST-CP maintains this vendor database as a focal point for environmental measurement technologies. The CMST Vendor Database matches user's measurements needs with available products.

H.7.3 The following websites provide a verification and evaluation assessment of several technologies. They represent an unbiased assessment of the technologies, normally performed by representatives of EPA or their contractors.

- EPA **ETV** (**E**nvironmental **T**echnology **V**erification) Program
  http://www.epa.gov/etv/
  This website provides credible environmental technology performance data for a variety of field analytical technologies resulting from an evaluation performed by independent third parties under the auspices of EPA. To date, ETV has generated several Verification Statements and Reports for several vendors' products for the following field analytical technologies: X-ray fluorescence, polychlorinated biphenyl (PCB) screening technologies (i.e., EPA/600/R-98/113, Immunoassay Kit: Strategic Diagnostics, Inc., EnviroGard PCB Test Kit), portable gas chromatography/mass spectrometry (GC/MS), soil gas sampling/analyses techniques, and SCAPS (site characterization and analysis penetrometer system). Future plans are to evaluate explosive screening tools.

- Cal/EPA California Environmental Technology Certification Program
  http://www.calepa.ca.gov/CalCert/
  Cal/EPA's certification program is a voluntary program that provides participating technology developers, manufacturers, and vendors an independent evaluation of the performance of new and mature environmental technologies. Performance claims made by the manufacturers are evaluated, and where necessary, additional testing is conducted to verify claims. The Web site provides access to the certification reports. Certifications may provide estimates of performance in areas such as efficacy and efficiency for specified uses, matrices, and chemicals; accuracy, precision, and detection limits for measurement of specified constituents; and other performance criteria. Currently, Cal/EPA has evaluated several monitoring technologies that are quicker and less expensive for detecting and measuring various contaminants (including BTEX (benzene, toluene, ethylbenzene, and xylenes), mercury, PCB, PCP (pentachlorophenol), PAHs (polynuclear aromatic hydrocarbons), petroleum hydrocarbons, TNT, and RDX) in contaminated soil and/or groundwater. Additional evaluations include accelerated solvent extraction (ASE) instrumentation, insitu subsurface field screening method for petroleum, oil, and lubricants that contain polynuclear aromatic compounds using laser-induced fluorescence (SCAPS-LIF), and a continuous on-line hydrocarbon monitor for waters.

- EPA **SITE** (**S**uperfund **I**nnovative **T**echnology **E**valuation) Reports
  http://www.epa.gov/ORD/SITE/index.html
  This website provides information on a wide variety of vendor-specific field analytical technologies for the demonstration and evaluation for use in the cleanup of Superfund Sites. Through the SITE Monitoring and Measurements program, the EPA National Environmental Research Laboratory - Las Vegas has produced several Innovative Technology Evaluation Reports/Profiles to document the results of field analytical technologies it has demonstrated.

H.7.4 The following websites provide information on the application of a field analytical technology to a particular environmental project. Depth of information varies widely from detailed memorandums to case study worksheets outlining general information on applicability, benefits, limitations, and costs.

- EPA **CLU-IN** (**CL**ean-**U**p **IN**formation)
  http://www.clu-in.org/
  This website provides information about innovative technologies for treatment, characterization, and monitoring of hazardous waste remediation projects. Several direct links are provided to key environmental programs and organizations, and others note points of contact or provide files that may be downloaded for access to project reports and memorandums. Numerous publications on the application of field analytical technologies to remediation projects are available for downloading. A few are highlighted below:

- Field Analytical and Site Characterization Technologies - Summary of Applications (EPA-542-R-97-011), November 1997.
- A Guideline for Dynamic Workplans and Field Analytics: The Keys to Cost-Effective Site Characterization and Cleanup (case study).
- Consortium for Site Characterization Technology Fact Sheet, 1997.
- Geophysical Techniques to Locate DNAPLs: Profiles of Federally Funded Projects, 1998.
- Improving the Cost Effectiveness of Hazardous Waste Site Characterization and Monitoring, 2000.
- Innovative Technology Evaluation Reports (see ETV site also).
- University of Connecticut - Guidelines for Applying Field Screening Methods in Conducting Expedited Site Investigations at Underground Storage Tank Sites in Connecticut, November 30, 1996.
- Site Characterization and Monitoring Technologies: Bibliography of EPA Information Resources.
- Uncertainty Management: Expediting Cleanup Through Contingency Planning, 1997.
- EPA Innovations in Site Characterization - Interim Guide to Preparing Case Studies, EPA-542-B-98-009, October 1998

- DOE **CMST-CP** (**C**haracterization, **M**onitoring and **S**ensor **T**echnology **C**ross Cutting **P**rogram) http://em-52.em.doe.gov/ifd/rbbooks/cmst/cmstrb.htm
  This website provides information on the application of several unique field analytical technologies (PAWS (portable acoustic wave sensor systems), SAWs (surface acoustic wave array detectors), portable GC/MS systems, etc.) that are focused on the characterization of mixed waste and high-level and environmental wastes.

H.7.5 Other sources of information include various Federal, State, local, and private organizations, publications, vendor Web sites, etc. A few examples are given as follows:

- Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM), EPA 402-R-97-016 (NUREG-1575), December 1997 (radionuclide field analytical technologies)
- EPA Innovations in Site Characterization - Interim Guide to Preparing Case Studies, EPA-542-B-98-009, October 1998
- EPA Region I, New England - Immunoassay Guidelines for Planning Environmental Projects, October 1996

- CMECC (California Military Environmental Coordination Committee) - Field Analytical Measurement Technologies, Applications, and Selection, April 1996

- ASTM Methods, Draft Standard Guide for Selection of Chemical Field Screening and Field Analytical Methods Used in Vadoze Zone Investigation

- Current Protocols in Field Analytical Chemistry, V. Lopez-Avila, ed., John Wiley & Sons, Inc., New York, 1998.

- Manufacturer/Vendor Information (paper and electronic)

Appendix I
Shell for Analytical Chemistry Requirements

I.1 Purpose and Use

The policy of the U.S. Army Corps of Engineers (USACE) is to produce data of known quality that meet all project-specific requirements established by the Technical Project Planning Team as outlined in EM 200-1-2. In order to meet those goals, ER 1110-1-263 requires that all analytical service providers have verifiable quality systems compliant with the principles of Guide 25 of the International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) (1990). Many of the components specified within this appendix are pursuant to those standards. However, refer to EM 200-1-1 for comprehensive guidance on the requirements needed to comply with this directive. In general, Sections I.1 through I.3 are for use by personnel generating the contract Scope of Work (SOW) or project Sampling and Analysis Plan (SAP), providing guidance on the establishment of project measurement quality objectives based upon the intended use of the data; and Sections I.4 - I.13 are for the analytical service providers or laboratory, providing guidance on implementation and interpretation of the policy and method requirements established herein. In addition, bold-face italicized type is utilized throughout the guidance to cue input based upon project-specific requirements, to clarify USACE project policy, or to identify when appropriate notification procedures to the client/data user are required. To support the application of this guidance to a project, it has been designed to allow a project team/chemist to apply the guidance as a whole or inappropriate sections by reference within a contract or SOW, using the laboratory measurement quality objectives as specified, or modify them (make them more or less stringent) based upon project requirements. USACE technical personnel should also review topics in bold-face type within applicable analytical sections to identify necessary project-specific information. These project-specific clarifications should then be discussed or summarized within the project documents (e.g., SOW and SAP) to avoid misunderstandings between the laboratory and USACE. Items included as recommendations also have the potential for enforcement as a requirement based upon project data quality objectives (DQOs). Summary tables are included within this guidance to outline the specific quality control (QC) items for each method and associated measurement quality objectives. This summary format is designed to facilitate the adjustment of QC parameters to reflect project-specific DQOs. It should be noted that these measurement quality objectives apply to the laboratory QC procedures exclusively, and do not address the field control sample QC requirements. The calculation of data quality indicators (DQIs) for laboratory precision and bias represents only the analytical error or an estimated 20 percent of the total error associated with each sample. For guidance on establishing field (quality assurance (QA)/QC) replicate precision requirements, refer to EM 200-1-6 and Grant, Jenkins, and Mudambi (1996).

I.1.1 USACE data needs.

I.1.1.1 USACE currently executes restoration activities under several environmental regulatory programs. The analytical testing of various environmental samples is often a significant part of these activities. The data must be produced by a process or system of known quality to withstand scientific and legal challenge relative to its intended purpose. To give the USACE programs the greatest flexibility in the execution of its projects, the SW-846 methods (U.S. Environmental Protection Agency (USEPA) are generally the methods employed for the analytical testing of environmental samples. The decision to focus on SW-846 methods is due largely to their being comprehensive for various media and chemical parameters; subsequent promulgation of method updates keeping methods current with instrument capabilities and industry standards; and their flexibility allowing adaptation to individual project-specific requirements. When SW-846 or other national standard methods are not available for the medium, methods published by reputable technical organizations (e.g., American Society for Testing and Materials (ASTM)) or refereed scientific journals should be pursued. The use of nonstandard methods shall be subject to agreement between

the laboratory and USACE. All nonstandard methods shall be fully documented within project-specific SAPs, and must undergo full review and approval procedures as outlined in the SAP.

I.1.1.2 The Shell for Analytical Chemistry Requirements establishes a basic approach for application of analytical chemistry methods (e.g., SW-846, performance-based methods) by the USACE. The concepts included here specify a baseline implementation of several analytical chemistry methods. However, when a performance-based analytical approach is employed, additional regulatory approval may be necessary to ensure acceptance of data generated.

I.1.2 Project chemical data quality management (CDQM).

I.1.2.1 All Hazardous, Toxic, and Radioactive Waste (HTRW) projects require a comprehensive program of CDQM activities to support the generation of data of acceptable quality. EM 200-1-6 outlines several QA techniques that may be applied to help evaluate the quality of sampling and analyses performed. USACE project team members must develop a project-specific CDQM program by defining appropriate monitoring techniques based upon the type of data generated and its intended purpose. The compliance monitoring techniques chosen for a project are then detailed within the project contracting documents (e.g., SOW, guide specification), if applicable. In general, data collection efforts involve the design of project plans to achieve the data quality objectives, which support progress toward site closeout; correct implementation of those project plans; and assessment of the data to determine if the data quality objectives were met. ***To ensure full accordance among the USACE, Architect-Engineer firms and laboratories, the project plans (i.e., SAP) shall discuss all of the project-specific compliance monitoring CDQM activities applied to the given project.*** For instance, compliance monitoring activities may include laboratory accreditation requirements; collection/analyses/evaluation of appropriate field and laboratory control samples; measurement quality objectives attainment/corrective action scenarios; external audits (field and/or laboratories); or external data assessment procedures (i.e., data validation, magnetic tape audits).

I.1.2.2 In order to promote flexibility as well as some degree of consistency in the data generated to support USACE HTRW projects, when inconsistent or mutually exclusive method requirements are encountered, the following hierarchy applies:

- Project-specific documents (e.g., SAP).
- USACE Engineer Manuals or other policy guidance.
- SW-846 methods.

Hence, the laboratory should be aware of and review these sources to determine project-specific DQOs and applicable project requirements.

I.1.3 Performance based methods implementation.

I.1.3.1 As the various Federal, State, and local regulatory agencies acknowledge the adoption of Performance Based Measurement Systems (PBMS) as a means to achieve required environmental monitoring, the applicability of performance-based methods to individual projects will increase. PBMS are defined by USEPA as a set of processes wherein the DQOs of a monitoring program are designated, rather than specifying the approved standard analytical method necessary. To date, however, the details for establishing data quality and performance requirements for required monitoring to support the assessment and selection of performance-based methods have not been fully defined within the various USEPA and State environmental offices. In addition, progress in updating Federal, State, and local regulations to incorporate

the PBMS philosophy and remove the requirements for specified standard reference methods for the use of new and innovative technologies is necessary to help assure successful PBMS implementation. Currently, PBMS has encouraged the application of field analytical technologies to environmental restoration projects.

I.1.3.2 This performance-based method approach empowers the analytical service (data) provider with the flexibility to vary aspects of an analytical system and protocols as long as the demonstrated method performance meets the requirements established by the data user(s). A PBMS may employ completely different chemistries or detection systems from those identified in current standard reference methods; may alter sample preparatory or determinative procedures that enhance or inhibit extraction/digestion or signal efficiency; or may encompass only minor modifications to a standard method instrument configuration. Due to this inherent flexibility, additional effort is necessary in the planning and executing phases to ensure successful implementation of performance-based methods. ***This may include any or all of the following: establishing and maintaining proper PBMS documentation (i.e., method standard operating procedures (SOPs), records of data analyses/results); USACE and regulatory agency review/approval; evaluation of method performance via DQIs; and comparison of PBMS data to data generated from a standard reference method.*** Before implementation of performance-based methods, the analytical service provider must establish the capabilities of the method/technique, to include selectivity, sensitivity, range of detection, precision, and bias. These are evaluated against performance criteria established by USEPA, State regulatory agencies, or the technical project planning team to assess the usability of the PBMS or performance-based method. ***The accuracy of the developers'/manufacturers' claims and technical data and the comparability amongst various techniques should be scrutinized for it is an area that is in the early stages of standardization by USEPA.*** In the event that the method capabilities do not meet project requirements, differences shall be reconciled prior to project execution. Reconciliation may require modifying the selected method, choosing an alternative method or techniques, or modifying the project DQOs. ***Project application of performance-based methods requires that performance be demonstrated for the analytes of concern, at the levels of concern in the matrix of concern within a specified acceptable error tolerance.*** Data generated from performance-based methods are evaluated using the same procedures as standard reference methods, as presented in Sections I-9, I-10, and I-11. In addition, if the PBMS is considered an emerging technology, lacks established records of use or application to environmental matrices, or varies significantly from the standard reference method, suggest acquiring a percentage of split samples for redundant analysis by the standard reference method. This will allow a comparison or calculation of a correlation factor between the data sets to evaluate the usability of the performance-based method in that project matrix.

I.2 SW-846 Methods Organization

I.2.1 SW-846 Methods implementation. EPA/SW-846 contains the analytical testing methods that the USEPA has evaluated and found to be acceptable for analysis of samples under Subtitle C of the Resource Conservation and Recovery Act (RCRA). As stated in the Final Rule that incorporated the Third Edition of SW-846 (and its updates) into the RCRA regulations, this publication is required to be used for only certain activities in the RCRA program. In other situations, this USEPA publication functions as a guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate, in satisfying RCRA-related sampling and analysis requirements. These methods are intended to promote precision, accuracy, low bias, sensitivity, specificity, and comparability of analyses and test results. SW-846 includes several separate test methods addressing hundreds of analytes. For any given analyte, multiple methods, with varying detection limits, are potentially available from this resource. As noted in Section I.1.1, USACE data needs focus on the use of SW-846 because the methods are comprehensive for many environmental matrices and chemical parameters, they are current with instrument capabilities and industry standards, and they are flexible to adaptation based on project-specific requirements.

I.2.2 SW-846 Method updates. SW-846 is a dynamic document that is subject to change as new information and data are developed. Advances in analytical instrumentation and techniques are continually reviewed by the USEPA Office of Solid Waste and Emergency Response and periodically incorporated into SW-846 updates to support changes in the regulatory program and to improve method performance. Any of these promulgated or draft SW-846 methods or other methods may be used by USACE to support the project-specific requirements. However, it should be noted that recent SW-846 updates have deleted several methods where technology was considered outdated (i.e., packed chromatographic columns) and incorporated several new field screening methods. Therefore, it is advisable to maintain current knowledge of these method advances and design projects, taking advantage of the most recently promulgated methods.

I.3 Project Objectives for Data Measurement

I.3.1 Data quality objectives (DQOs). To generate data that will meet the project-specific requirements, it is necessary to define the types of decisions that will be made and to identify the purpose of the data. DQOs are an integrated set of specifications that define data quality requirements based on the intended use of the data. Project-specific DQOs are established to encompass both field and laboratory operations. The DQO process leads to the specification of the following at a minimum: sample handling procedures; preparatory (extraction/digestion), cleanup, and determinative methods; target analytes; method quantitation or reporting limits; field and laboratory quality control samples; measurement quality objectives (QC acceptance limits) for DQIs (formerly precision, accuracy, representativeness, comparability, and completeness (PARCCS) parameters); required corrective actions; and data assessment procedures necessary to meet the intended use of the data. Special considerations that may also apply include internal laboratory sample chain-of-custody, data confidentiality, data archival, or data retention requirements beyond those stated herein. In the generation of the SAP, whether this is done by contractor or USACE personnel, a number of steps must occur at the planning stages for each project phase by various technical disciplines to identify all necessary data needs to formulate the project DQOs that support the project through close-out. Each project phase will have different information requirements and therefore different data needs. However, the project strategy as a whole must be kept in mind to optimize each phase, streamlining the project whenever possible to avoid repetitive sampling and analytical work efforts. These planning exercises will then lead to a comprehensive sampling and analytical protocol for each phase. These tasks may be performed by USACE for work done in-house, prescribed for a contractor within a Scope of Work, or developed by the contractor directly into the SAP. Refer to EM 200-1-2 for information on the development of these goals and objectives for data collection design.

I.3.1.1 Assessment of data needs. As presented in EM 200-1-2, data needs are determined for the project based upon the decisions that need to be made. At the same time, a determination of the data quality required for each piece of data (data need) must also be defined by the eventual data user. This information, routinely expressed as an uncertainty allowed or a qualitative statement of requirements, will help other technical planners (data implementors) to identify applicable sampling and analytical protocols to generate the required data. The data needs quality requirements are assessed by analytical parameter (per matrix, per area). Since it is possible to have more than one data user requesting the same data need(s), the most stringent data user requirements are applied to ensure the suitability of these data by all requesting parties. This information is then used to decide an appropriate analytical strategy to generating the required data. The analytical strategy may use a combination of screening and/or definitive data to support field and project decisions.

I.3.1.2 Assessment of data collection options. Initially, the applicability of field analytical methods to the objectives of the project should be investigated. The rapid or real-time results help support field decisions to guide or direct sampling and analytical activities. Thereby streamlining progress toward site end goals. Field analytical methods include qualitative or semiquantitative field screening techniques (e.g., photoionization detector/flame ionization detector, immunoassay, colorimetric, etc.), and quantitative onsite

techniques whose preparatory process and/or QC elements are typically less rigorous than those established for definitive data (e.g., X-ray fluorescence, gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), etc.). Standard analytical methods producing definitive data must also be reviewed for applicability to the project. Input necessary to determine applicable elements for both screening and definitive analytical techniques used include the following at a minimum: defining the contaminants of concern; the concentration range of interest; sensitivity requirements; measurement quality objectives for precision, bias, and completeness; the need and/or type of confirmation necessary; and whether any physical, chemical, or logistical constraints are germane. The appropriate methods may also be dictated by the data user (e.g., outlined by regulatory authority or record of decision.

I.3.2 Measurement quality objectives. To ensure that quality data are continuously produced during analysis and allow the eventual compliance review, systematic QC checks are incorporated into the sampling and analyses to show that procedures and test results remain reproducible and that the analytical method is actually measuring the quantity of target analytes without unacceptable bias. Systematic QC checks include the scheduled analyses of field and laboratory replicates, standards, surrogates, spiked samples, and blanks. Measurement quality objectives (acceptance criteria or ranges) for these systematic QC checks are established to verify DQIs support data usability and contract compliance. The program of systematic QC checks may be viewed from two aspects, batch QC and matrix-specific QC, and are further clarified below.

I.3.2.1 QC checks of known composition samples. General batch QC may be viewed as those QC procedures applied to an interference-free matrix or a matrix of known composition (i.e., blanks, laboratory control samples, single or double-blind performance evaluation (PE) samples, standard reference materials, calibration verification standards, etc.). They ensure the sampling procedures are appropriate and the analytical method are being performed in an in-control mode of operation. These QC checks provide no information on how well the method is performing with respect to the project sample matrix, however. QC checks that exceed measurement quality objective must be clearly documented within the case narrative the along with corrective actions taken. ***It is recommended that contract nonpayment clauses be limited to QC sample results of interference-free or known composition matrices only. An example of a contract nonpayment clause that may be included within project contract documents follows:***

> "The Contractor shall perform chemical analyses in accordance with the requirements established within the specified method and this appendix. When QC checks of an interference-free or known composition do not meet these standards/requirements, corrective action must be taken through proper application of the inspection and services clause. Corrective action may include resampling, repreparation, and/or reanalyses of the affected samples at no additional cost to the Government. If the Contractor fails to promptly perform the required corrective actions, or when the failure cannot be corrected by reperformance, the Government may reduce the contract price or fee payable under the contract to reflect the reduced value of services performed. Continued failure to perform chemical analyses in accordance with these standards/requirements may result in termination of the contract for default."

I.3.2.2 QC checks of matrix-specific samples.

I.3.2.2.1 Matrix-specific (matrix-sensitive) QC procedures should be incorporated into the sampling and analysis protocols to provide information on the precision and bias of the analyses on project samples. These procedures include analyses of field samples in association with surrogate compounds, matrix spikes (MS), matrix spike duplicates (MSD), or matrix duplicates (MD). Matrix-specific procedures performed on other field samples at the laboratory not associated with the project samples are of no value, for they do not provide information on the matrix under observation. It should be noted that MS/MSD/MD analyses may require the submittal of an additional replicate sample to enable the laboratory adequate sample volumes to

perform the requisite analysis. ***For this reason, the project requirements of minimum sample volumes necessary to accommodate the matrix-specific QC samples must be addressed very clearly within the SAP.***

I.3.2.2.2 Exceedances of measurement quality objective for these types of QC checks may be problematic due to matrix effect (signal enhancement or suppression) on the analysis and should not be viewed as an indicator of poor laboratory performance. For this reason, ***contract nonpayment clauses should not be associated with matrix-specific QC samples.*** However, the laboratory should not use this as an "excuse" to avoid employing proper analytical techniques. The laboratory should make a reasonable effort to overcome matrix interference as noted later in this paragraph. Necessary corrective actions will vary depending on the type of interference, and are subject to analyst professional judgment. ***When these excursions indicate a potential for false negatives, lack of sensitivity, or an inability to accurately detect the target analytes, communication between the laboratory and data user should be pursued to identify alternatives available.*** For instance, procedures to decrease the matrix effect may include implementing cleanup procedures, dilution techniques, smaller sample size processed, etc. However, consequences to the data (i.e., higher detection limits, less representative aliquot, etc.) should also be assessed against project objectives.

I.3.3 Data quality indicators (DQIs). As previously noted, QC procedures are employed during chemical analysis to support and document the attainment of established measurement quality objectives. Whether these QC procedures support an assessment of general batch control or matrix-specific application, documentation includes calculating DQIs to verify data usability and contract compliance. DQIs were formerly referred to as the PARCCS parameters of precision, accuracy, representativeness, comparability, completeness, and sensitivity. All laboratories conducting analytical work for USACE must be aware of, and be in agreement with, the project DQOs, including the stated DQI - measurement quality objectives. ***To avoid any misunderstandings concerning the level of quality required for the project chemical analyses, the SAP must very clearly delineate all measurement quality objectives for the method QC checks and DQIs (precision, bias, representativeness, comparability, completeness, and sensitivity) for each method applied.*** Tables I-1 through I-8 summarize the measurement quality objectives for eight SW-846 methods. ***These tables may be applied directly to a project, or modified accordingly to define the measurement quality objectives for laboratory DQIs (precision (P) and bias (B)) of the laboratory control samples (LCS), MS, MD/MSD, etc. However, project requirements must still be defined for the remaining applicable DQIs within appropriate project documents (e.g., SOW, SAP), for example, DQI performance objectives of field QC samples (precision objective for field replicates, bias objective for field blanks, bias (accuracy) objective of double-blind PE samples, etc.); DQI performance objectives for matrix-specific sensitivity (per requisite methods); DQI performance objectives for project completeness (note whether field and lab completeness are assessed separately or combined); and qualitative DQIs (representativeness, comparability).***

I.3.3.1 Precision. Precision refers to the distribution of a set of reported values about the mean, or the closeness of agreement between individual test results obtained under prescribed conditions. Precision reflects the random error and may be affected by systematic error. Precision also characterizes the natural variation of the matrix and how the contamination exists or varies within that matrix. For chemical parameters that do not allow homogenization prior to sample acquisition (e.g., volatile organic analysis), precision values must be reviewed accordingly. ***In order to assess the effect these variables have on the total precision of data, both field and laboratory replicates should be acquired.*** In order to assess matrix heterogeneity or sample handling procedures, field precision is commonly determined from field duplicate samples or quality assurance split samples. For environmental samples, laboratory precision is commonly determined from laboratory duplicate samples (i.e., MS/MSD or MD samples). However, to establish the precision of a given analytical method without the effect of a matrix, a laboratory control sample is necessary. USACE currently recommends the inclusion of a laboratory control sample duplicate within a batch, but does not require it. Statistical measures of precision include relative percent difference (RPD)

(relative range for duplicates), standard deviation, or relative standard deviation. The RPD for a set of duplicate measurements of a variable X is defined as:

$$\mathbf{RPD} = \left\{ \frac{|X_1 - X_2|}{[(X_1 + X_2)/2]} \right\} \times 100\% \tag{I-1}$$

If sufficient replicates are taken from a particular matrix for a project, precision may be expressed as the standard deviation, Percent Relative Standard Deviation (RSD), or Coefficient of Variation (CV). This value assesses the precision of the sample within that population matrix, where n is the number of samples or data:

$$\mathbf{RSD} = \mathbf{CV} = \left(\frac{[\text{std dev}]}{\bar{X}} \right) \times 100\% \tag{I-2}$$

$$[\text{std dev}] = \left[\Sigma (X_i - \bar{X})^2 / (n - 1) \right]^{1/2}$$

I.3.3.2 Bias. Bias refers to the systematic or persistent distortion of a measurement process that causes errors in one direction (above or below the true value or mean). Bias may be affected by errors made in field or laboratory handling procedures. For example, procedural deviations in sample acquisition, incomplete homogenization prior to subsampling, or incomplete extraction of contaminants from the matrix intensifies bias. Bias is a term that is related to but is not interchangeable with accuracy. ***Bias assessments are typically based upon the analysis of spiked reference materials or spiked samples (i.e., LCS, MS, MSD, surrogates).*** When the sample matrix is spiked, the result allows an assessment of the effect of the sample matrix on recoveries. The sources of error contributing to the bias of a measurement can be difficult to determine for an entire sample collection/analysis activity. Sources of error may include the loss (or addition) of contaminants from the sampling and analysis process (i.e., sample handling, field cross-contamination, improper sample preservation, sample manipulation during preparation and analysis), interferences present within the sample matrix, and measurement error (i.e., calibration error or drift). Bias values for the LCS represent quantitative limits beyond which data are unacceptable. Bias values are commonly expressed as percent recovery. Percent recovery (%R) is calculated as follows:

$$\%R = \left[\frac{(X_s - X_u)}{K} \right] \times 100\% \tag{I-3}$$

where

X_s = measured value of the spiked sample

X_u = measured value of the unspiked sample

K = known amount of the spike in the sample

When %R is calculated for LCS or other reference materials, X_u could be set at zero. The relationship between percent bias % B and percent recovery is as follows:

$$\%B = \bullet \%R - 100 \bullet \tag{I-4}$$

I.3.3.3 Accuracy. Accuracy is the measure of the closeness of an observed value to the "true" value (e.g., theoretical or reference value, or population mean). Accuracy includes a combination of random error and systematic error (bias) components that result from sampling and analytical operations.

I.3.3.4 Representativeness. Representativeness refers to the degree to which sample data accurately and precisely describe the characteristics of a population of samples, parameter variations at a sampling point, or environmental condition. Samples that are not properly collected or preserved (e.g., contaminant loss or addition) or are analyzed beyond acceptable holding times should not be considered to provide representative data. ***Representativeness is a parameter that is concerned primarily with the proper design of the sampling program or subsampling of a given sample.*** An assessment of representativeness would include an evaluation of precision. The representativeness criterion is best satisfied in the laboratory by making certain that all subsamples taken from a given sample are representative of the sample as a whole. This would include sample premixing/homogenizing prior to and during aliquotting procedures. Samples requiring volatiles analysis should not undergo any premixing or homogenization. Therefore, noting sample characteristics in a case narrative may assist in the evaluation of data. Representativeness can be assessed by a review of the precision obtained from the field and laboratory duplicate samples. In this way, they provide both precision and representativeness information. Existing project data and geostatistics may be employed to assess the representativeness of a population by defining the continuity of data from point to point. Geostatistical techniques can then be used to predict spatial distribution of contaminants, aid in the development of future project sampling designs, identify sample locations, optimize sample spacing, estimate probabilities, etc. Applicability of representativeness in assessing a contaminant population is improved by using a larger number of samples.

I.3.3.5 Comparability. Comparability is a qualitative objective of the data, expressing the confidence with which one data set can be compared with another. Sample data should be comparable for similar samples and sample conditions. ***This goal is achieved through the use of standard techniques to collect representative samples, consistent application of analytical method protocols, and reporting analytical results with appropriate units.*** Comparability is unknown unless precision and bias are provided. When this information is available, the data sets can be compared with confidence. ***When PBMS methods (i.e., new or modified standard reference methods or field analytical techniques) are employed, comparability becomes a critical and potentially quantitative data quality indicator.*** As noted in Section I.1.3, PBMS methods may employ significant differences from the standard reference method used for that same target analyte or chemical compound class. ***If comparability with standard methods has not been demonstrated, a project-specified percentage of duplicate (split) samples for analysis by the standard reference method should be included.*** This allows an assessment of comparability between data sets by calculating the RPD (or a correlation factor adjustment), thus determining the usability of the performance-based method in supporting project decision making. ***Further recommend establishing (and documenting in the project SAP) a project-specified comparability acceptance criterion (e.g., RPD • 30 - 50 percent, assuming a one-to-one correlation) based on the intended use of the data and project objectives.***

I.3.3.6 Completeness. Completeness goals, if defined for individual sampling and analytical protocols, are normally combined to assess the expectations of the project as a whole. Completeness is the percentage of measurements that are judged to be usable (i.e., which meet project-specific requirements) compared to the total number of measurements planned. ***Specified levels of overall (both field and laboratory) completeness, in addition to particular completeness goals for critical samples, should be set as part of the project DQOs in the project SAP.*** It is important that critical samples are identified and appropriate QC maintained to ensure that valid data are obtained in order to secure the requisite type, quantity, and quality of data necessary to complete the project. The desired level of completeness is dependent on the project-specific DQOs. This information will be conveyed to the laboratory within the SOW or project SAP. Planning and communication among all parties involved in the process are crucial in order to achieve high completeness percentages. However, completeness goals of 100 percent are usually unattainable. ***Realistic completeness goals (i.e., 80-95 percent) should be determined based upon the size and complexity of the project.***

I.3.3.7 Sensitivity. The term sensitivity is used broadly here to describe the contract method detection/quantitation/reporting limits established to meet project-specific DQOs. Several limits have been established to describe project sensitivity requirements, such as IDL (instrument detection limits), MDL (method detection limits), PQL (practical quantitation limits), SQL (sample quantitation limit), CRDL (contractor-required detection limits), CRQL (contractor required quantitation limits), etc. The IDL and MDL are considered minimum values for detection for the instrument only, and for the instrument and sample preparation steps, respectively. Both the IDL and MDL are normally based on an interference-free matrix (i.e., reagent water or purified solid), which ignore sample matrix effects on those limits. For this reason, published MDLs or IDLS are presumably not achievable for environmental samples. The PQL is defined as the lowest level that can be reliable measured by routine laboratory operating conditions within specified limits of precision and accuracy. This is considered equivalent to the Method Quantitation Limit as defined in section I.3.3.7.2. The SQL is a term established within the USEPA Risk Assessment Guidance for Superfund (RAGS), and are the limits of interest for reporting data for use in a risk assessment. The SQL is defined as the MDL adjusted for sample-specific action such as dilution or use of varying sample aliquot sizes. The CRDL and CRQL published within Contract Laboratory Program (CLP) methodologies are contractually based levels and do not pertain to instrument sensitivity.

I.3.3.7.1 Method detection limit (MDL). The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. A laboratory shall, at a minimum, perform MDL studies during initial method setups and whenever the basic chemistry of the procedures is changed. The MDLs shall be preparatory method-specific and include any cleanup methods used. Since it is not practical to establish an MDL for each specific matrix received at any given laboratory, MDLs shall be determined for all target analytes in an interference-free matrix, typically reagent water for aqueous samples, and a purified solid matrix (e.g., sand) for soil/sediment samples. The laboratory may determine MDLs using procedures presented in 40 CFR, Part 136, Appendix B, or equivalent statistical approach. The validity of the MDL study is verified per CFR requirements by comparing the analyte values to the calculated MDL. If the analyte values are below the calculated MDL or greater than ten (10) times the calculated MDL, an unacceptable bias may be induced; and the MDL cannot be reported. ***To ensure that valid MDL values are determined, the laboratory shall analyze an MDL check sample by spiking an interference-free matrix with all target analytes at about two times the calculated MDL.*** The MDL check sample shall be taken through all the preparatory and determinative steps used to establish the calculated MDL values, to verify a response is detected. If any of the target analytes are not detected, then the concentration shall be increased in another MDL check sample, and the analysis repeated until the failed target analytes are detectable. The detectable target analyte concentrations shall then be used in lieu of the calculated MDL values to establish the lowest detected concentration for samples taken through all appropriate method procedures. ***The laboratory may then demonstrate continued method detection capability by analyzing the MDL check sample on a quarterly basis, in lieu of the annual MDL study.*** When multiple instruments or confirmation columns are used for the same method, separate MDL studies may be replaced by the analysis of an MDL check sample on all instruments/columns. The MDL check sample shall be analyzed after major instrument maintenance or changes in instrumentation or instrumental conditions to verify the current sensitivity of the method. ***When low-level detection in a project matrix is critical, it is suggested that the laboratory perform a method detection limit study or an MDL check sample in project-specific matrices. This should be done at project start-up in order to accurately assess the detection capability of the method for project samples.***

I.3.3.7.2 Method quantitation limit (MQL). Due to the significant amount of error (approximately ±100 percent) associated with results calculated at the MDL and the fact the MDL may not be attainable within project matrices, the method quantitation limit (MQL) is established at a factor of five to ten times

the MDL for the majority of target analytes, but no lower than three times the MDL for any target analyte. The statistical error (±20-30 percent) associated with this area of the calibration curve is notably reduced from the MDL. The appropriate factor applied to the MDL to establish the MQL is based upon the acceptable amount of error the data user is willing to accept for the data generated. Ideally this MQL should have an associated error comparable to the method-prescribed continuing calibration verification (CCV) acceptance limits. This may not be feasible, however, due to a lower concentration range of interest. This approach, however is not appropriate for multicomponent target analytes. Due to the identification of multicomponent target analytes (e.g., polychlorinated biphenyls (PCBs), chlorodane, toxaphene, gasoline, etc.) being based upon a recognizable pattern, the MQL should be based upon the MDL as well as the concentration at which the pattern is reliably "identifiable". Thus the MQL represents the value at which the laboratory has demonstrated the ability to reliably quantitate target analytes within a prescribed performance criterion for the method, and establishes the lowest concentration at which the data may be reported without qualification as an estimated value (i.e., J-flag). ***In the absence of project-specific requirements to the contrary, USACE requires the following:***

- ***The MQL is set at the lowest standard used for the initial calibration curve (or low-level calibration verification standard) or higher for each target analyte. The lowest standard or low-level calibration verification standard must be at least three times the MDL or greater.***

- ***Target analyte values detected and reported below the MQL must be flagged as an estimated quantity (i.e., J-flag).***

I.3.3.7.3 Method reporting limit (MRL). The method reporting limit (MRL) is a threshold value below which the laboratory reports a result as non-detected (U); and must be presented as "less than a concentration value" (< ##) according to the following. When data is used explicitly to support a risk assessment, the MRL must be equivalent to the SQL, which establishes the lowest reporting limit at the MDL adjusted for sample actions (dilutions/concentrations). However, when the data is being used for other purposes that cannot tolerate a high degree of error (including FNs and FPs), the lowest reporting limit must be established following method procedures defined in I.3.3.7.1. This includes data used for remedy implementation, confirmation, no further action (NFA), or site closure. The highest value reported for the MRL is dependant upon project-specified action or decision levels and is discussed in detail in the following section. MRLs are adjusted based on the sample matrix and any sample dilutions/concentrations necessary.

I.3.3.7.4 Project MRLs relation to action levels. When defining appropriate contract requirements for the MRL, the following issues should be considered.

- ***The data needs identified by the data user***

- ***The uncertainty associated with the low-level data that the user is willing to accept***

- ***The sensitivity capability of the method and instrumentation***

The data needs may be associated with compliance issues, establishing action or decision levels for the project. For instance, maximum contaminant levels (MCLs), maximum contaminant level goals (MCLGs), media cleanup standard, or other applicable, or relevant and appropriate requirements. Other data users (e.g., risk assessor) may require a toxicity reference concentration, a preliminary remediation goal, or other concentration of interest. ***In general, USACE recommends that the MRLs be established at approximately one-half the expressed project action levels.*** This establishes an acceptable degree of confidence in the resulting data to minimize the potential for false positive (type I) and false negative (type II) decision errors in the comparison of data near the stated project action levels.

The range of MRL determinations to support non-risk related data uses is shown in Figure I-1.

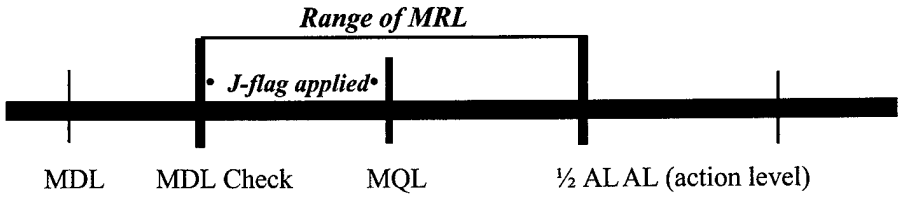


Figure I-1. Range of MRL determinations to support non-risk related data uses

The range of MRL determinations to support a risk assessment data use is shown in Figure I-2.

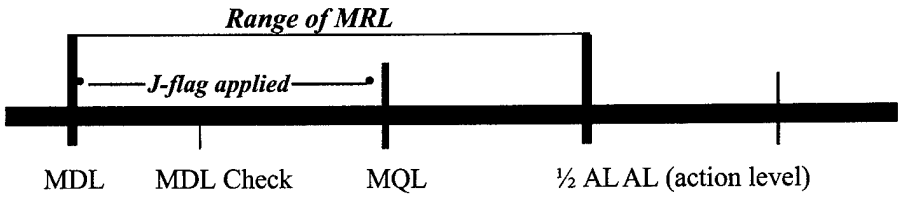


Figure I-2. Range of MRL determinations to support a risk assessment data use

- ***The project-specific MRLs may be established anywhere in the range from the MDL check sample concentration (two times the MDL) or MDL (adjusted for sample action) up to one-half of the project action level.***

- ***When the project-specific MRLs are established below the laboratory MQL for that method, the sample results reported (below the MQL) will be qualified as estimated. If very low levels of quantitation are required (e.g., data used for a risk assessment or compliance issue), to avoid estimation of data based upon this requirement, the following is recommended. Analyze a low-level check sample (taken through the appropriate preparatory procedures) at the MRL level to assess the accuracy at this concentration. An appropriate performance criterion should be defined based upon this assessment.***

- ***If the project action levels are near or below the MDLs generated by the laboratory or proposed within the methods, it is unlikely the sensitivity of the method will be achievable for an environmental matrix without imposing method variations. Discussions with USACE or laboratory personnel may be needed to identify options available to lower the MDL of the method (i.e., increase initial sample volumes, decrease final extract volumes, increase sample extract analyzed/injected, etc.), or to select an alternative method.***

- ***If the recommended factors cannot be accommodated, the data user is informed of this predicament, and a compromise must be reached. Compromise may entail accepting a higher degree of error associated with the data reported near action levels, or the acceptance of a higher potential for false negatives (type II error) near the MDL.***

I.4 General Laboratory Requirements

Per ER 1110-1-263, each laboratory performing work for USACE shall comply with ISO/IEC Guide 25 (1990). This may be accomplished by the application of the USACE laboratory validation as identified in

ER 1110-1-263. Procedures for the laboratory validation process are described in EM 200-1-1. The following laboratory requirements are pursuant to meeting the standards established within the noted references. ***Individual project requirements may be more or less stringent than those described in the following sections.***

I.4.1 Laboratory quality system. A laboratory must establish, implement, and maintain a quality system appropriate for the type, range, and volume of analytical services it provides. The elements of this quality system shall be documented within a Laboratory Quality Management Plan (LQMP) or related documentation. Laboratory management is responsible for communicating the stated policies and practices to laboratory personnel, ensuring all information is clearly understood and implemented. The laboratory shall perform periodic audits of activities to verify compliance with the quality system. When deviations are discovered, the laboratory shall take immediate corrective action to remedy the situation or practice, notifying any client whose work may have been affected.

I.4.2 Laboratory quality management plan (LQMP). The laboratory shall prepare a written Quality Management Plan, which describes the general and specific procedures used within the laboratory to achieve scientifically valid and legally defensible data. ***This documentation requirement pertains exclusively to the laboratory and is not considered equivalent to the Quality Assurance Project Plan (QAPP), which is an integral part of the project-related SAP.*** However, the laboratory may be required to submit this documentation as an appendix to the project-specific QAPP. ***When conflicting language exists between the project QAPP and the LQMP, the project QAPP takes precedence over the LQMP.*** The Quality Management Plan should present the policies, organization, objectives, functional guidelines, and specific QA and QC activities of the laboratory designed to achieve the data quality requirements when running performance-based methods, such as the SW-846 methods. SOPs pertaining to each element shall be included or referenced as part of this QA Management Plan and should describe the specific operational and analytical procedures as normally implemented by the laboratory. This plan should include, at a minimum, the following elements:

- QA policy, objectives, and commitments, any allowable departures from documented policies.
- Organization structure and personnel — include descriptions of key personnel, identify relationship between management, operations, support, and QA personnel.
- Facilities and equipment.
- Document control — notebook policy, sample tracking and custody procedures, LQMP and SOP organization and control.
- Scope of analytical methodologies provided — sample preparatory and determinative procedures available; methods implementation/calibration procedures and frequency, standards preparation procedures, traceability of measurements and procedures employed, decision processes/ procedures/responsibility for initiation of corrective action.
- Data generation — data collection procedures, data reduction procedures, data evaluation procedures, data reporting/authorization procedures.
- Quality control — solvent/reagent checks, reference material analysis, internal QC checks, retesting or corrective action implementation, verification of electronic data management systems.

- QA — determination and monitoring of method QA performance, systems/internal audits, customer complaints resolution, performance/external audits, interlaboratory comparisons and proficiency programs, corrective action procedures, and QA reporting procedures.

Submission of this Laboratory QA Management Plan for review, along with some or all of the SOPs, may be required before sample testing can be initiated on any given project. These documents shall be amended should deficiencies be noted during review or whenever the fundamental elements described previously are updated (i.e., annually).

I.4.3 Laboratory organization, management, and analytical personnel responsibilities. The laboratory shall have sufficient personnel with appropriate education, current training, and experience to fulfill their assigned duties. The laboratory shall promote independence of judgment and integrity with well-defined responsibilities outlined for each individual within the laboratory organization. Personnel training records shall be maintained by the laboratory.

I.4.3.1 Laboratory management. Laboratory management shall at a minimum have a technical director/manager responsible for overall technical operations. The technical director shall have a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline, and a minimum of 2 years of laboratory experience. The laboratory management shall have sufficient authority and resources to fulfill their duties accordingly. Management staff shall be responsible for actively supporting the following at a minimum: implementing the policy and practices defined within the LQMP, maintaining accurate SOPs and enforcing their use in the laboratory, participating in interlaboratory comparisons and proficiency testing, certifying that personnel performing all tests have proper education and training, providing appropriate management and supervisory support to ensure adequate supervision of technical staff, providing a contingency plan that identifies backup personnel for key laboratory positions (i.e., technical director/manager, QA officer/manager, etc.) in the event of personnel absence, having policy and procedures in place that ensure protection of clients' confidential information and proprietary rights, and maintaining a work environment that emphasizes the importance of data quality.

I.4.3.2 Laboratory quality assurance officer. The laboratory shall at a minimum have a QA officer/manager, responsible for the laboratory quality system. The laboratory QA officer shall be responsible for maintaining the quality system and overseeing the QA aspects of the data. The QA officer shall work independently of the laboratory production management and have direct access to the highest level of management for decisions on laboratory policy and resources. In laboratories with limited staff (i.e., <10 technical personnel) the QA officer may also perform duties as the technical director or deputy technical director. QA officer shall at a minimum serve as a focal point for QA issues, perform oversight and QA review for all nonconformance reports, perform QA review for a percentage of laboratory analytical batches or project data packages, evaluate data objectively, independent of laboratory management influence, possess a general knowledge of the methods for which data review is performed, conduct internal audits on the entire technical operation annually, and monitor laboratory method performance by control charts/ranges evaluation, promoting method improvements as necessary. This individual should have a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline and be familiar with all laboratory operations. A minimum of 3 years of laboratory experience, including at least 1 year of applied experience with QA principles and practices in an analytical laboratory, is required. In addition, a working knowledge of general statistical concepts is recommended to support data review and method performance monitoring responsibilities.

I.4.3.3 Organic chemistry section. If applicable, the laboratory shall maintain an Organic Chemistry Section with appropriate personnel, facilities, and instrumentation to conduct the work required. The following disciplines must be clearly represented and staffed as project testing dictates.

I.4.3.3.1 Organic chemistry section supervisor(s). The GC/MS, GC, or Sample Preparation Laboratory Supervisors are responsible for all technical efforts of their respective laboratories, providing sufficient oversight of activities to ensure that data meet all terms and conditions expressed for the project. These individuals shall possess documentation supporting demonstration of performance for all areas for which they provide supervision. In addition, they should have a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline and a minimum of 3 years of laboratory experience, including at least 1 year of supervisory experience.

I.4.3.3.2 GC/MS analyst. Qualifications for these individuals should be a minimum of 1 year of experience in operating and maintaining GC/MS with a bachelor's degree in chemistry or in any related scientific/engineering discipline, or in lieu of the bachelor's degree, 3 years of experience in operating and maintaining the GC/MS and interpreting GC/MS data.

I.4.3.3.3 Gas chromatography (GC)/high performance liquid chromatography (HPLC) analyst(s). Qualifications for these individuals should be a minimum of 1 year of experience in operating and maintaining GC/HPLC equipment with a bachelor's degree in chemistry or a related scientific/engineering discipline, or in lieu of the bachelor's degree, 3 years of experience in operating and maintaining the GC/HPLC and interpreting GC/HPLC data.

I.4.3.3.4 Extraction/concentration technician. Qualifications for these individuals should be a minimum of a high school diploma and 1 year of college general chemistry. These individuals should also have a minimum of 1 year of experience in extraction/concentration.

I.4.3.4 Inorganic chemistry section. If applicable, the laboratory should maintain an Inorganic Chemistry Section with the appropriate personnel, facilities, and instrumentation to conduct the work required for the project. The following disciplines must be clearly represented and staffed as project testing dictates.

I.4.3.4.1 Inorganic section supervisor(s). The metals, wet chemistry, or sample preparation laboratory supervisor(s) is responsible for all technical efforts of the respective laboratories, providing sufficient oversight of activities to ensure that data meet all terms and conditions for each project. These individuals shall possess documentation supporting demonstration of performance for all areas for which they provide supervision. In addition, they should have a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline and a minimum of 3 years of laboratory experience, including at least 1 year of supervisory experience.

I.4.3.4.2 Inductively coupled plasma (ICP) analyst. Qualifications for these individuals should be a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline with 1 year of experience in operating and maintaining ICP instrumentation, or in lieu of the educational requirement, three additional years of experience in operating and maintaining ICP instrumentation.

I.4.3.4.3 Atomic absorption (AA) analyst. Qualifications of these individuals should be a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline with 1 year of experience in operating and maintaining AA instrumentation for graphite furnace, flame, and cold vapor AA, or in lieu of the educational requirement, three additional years of experience in operating and maintaining AA instrumentation, including graphite furnace, flame, and cold vapor techniques.

I.4.3.4.4 Inorganic sample preparation technician. Qualifications for these individuals should be a minimum of a high school diploma and a college level course in general chemistry or equivalent. These individuals should also have a minimum of 1 year of experience in sample preparation in an analytical laboratory.

I.4.3.5 Wet chemistry analyst. If applicable, qualifications of these individuals should be a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline. These individuals should also have a minimum of 1 year of experience with classical chemistry laboratory procedures in conjunction with the education qualifications, or in lieu of the educational requirement, 2 years of additional equivalent experience.

I.4.3.6 Radiochemical techniques analyst. Qualifications of these individuals should be a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline with 1 year of experience in performing radiochemical analyses, or in lieu of the educational requirement, three additional years of experience in operating and maintaining radiochemical instrumentation.

I.4.3.7 Technical staff backup. The laboratory should have a minimum of one chemist available at any time as a backup technical person for each analytical area to ensure continuous operations and accomplish the work required. These individuals should have similar education and experience requirements to the primary analyst.

I.4.3.8 Sample custodian and data management. The laboratory should also maintain and staff support positions for Sample Custodian and Data Management personnel. Qualifications for these individuals should be at a minimum of a high school diploma and appropriate on-the-job training.

I.4.4 Laboratory facility and equipment.

I.4.4.1 Laboratory facility requirements. The laboratory shall provide a secure testing facility that can accommodate the proper performance for the type, range, and volume of analytical services it provides. Facility entries must be controlled and monitored as necessary to assure restricted access, especially for areas affecting the quality of activities or data. The design of the facility must provide effective separation of incompatible testing activities and adequate energy sources, lighting, heating/cooling, and ventilation to ensure stability of voltage, temperature, humidity, or other pertinent environmental conditions. This may involve inclusion of an area under positive pressure for analysis of volatile organic compounds (VOC). Adequate monitoring of environmental conditions and general housekeeping should be maintained to avoid any influence on the testing activities performed.

I.4.4.2 Laboratory equipment requirements. The laboratory shall provide sufficient equipment, instruments, and related supplies for proper performance of work. All equipment used shall be reflective of the measurement accuracy necessary. The laboratory shall ensure that all equipment and supplies purchased are inspected, a unique identifier assigned to it, and the equipment verified as compliant with all relevant requirements prior to their initial use. Records of all suppliers used to obtain support services and materials shall be maintained.

I.4.4.2.1 Equipment preventive maintenance. To minimize downtime and interruption of analytical work, preventive maintenance shall be routinely performed on each analytical instrument. Designated laboratory personnel should be trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, the equipment shall be taken out of service, repairs performed by either trained staff or trained service engineers, and an evaluation of the impact on previous calibrations or tests performed. It is generally recommended that maintenance contracts be maintained on all major analytical instruments. Detailed SOPs shall be on file or the information incorporated into method SOPs/LQMP that describe preventive maintenance procedures and schedules. The laboratory shall maintain detailed logs for each instrument documenting the preventive maintenance and repairs performed.

I.4.4.2.2 Equipment backup capabilities. Backup instruments shall be designated in case of an extended breakdown for an analytical instrument. It is the laboratory's responsibility to have a backup plan in force to ensure that all sample holding times can be met. This plan can include rental of backup instruments or the use of another USACE validated laboratory for a given procedure. All equipment outside of the laboratory's permanent control shall be evaluated to ensure that all relevant requirements are met prior to its initial use. ***Before any subcontracting is performed, USACE must be informed and approval given, in writing.*** The laboratory shall ensure, and be able to document, that all subcontractors employed are competent to perform the duties requested and comply with all of the requirements established within this guidance and EM 200-1-1, as appropriate.

I.4.4.2.3 Laboratory equipment records. The laboratory shall maintain appropriate records or documentation for all instruments and support equipment to identify type of equipment; manufacturer's name or equipment make, model, and any serial numbers or unique identifiers; dates received and placed into service; condition when purchased (new, used, etc.); current location; manufacturer instructions/manuals; history of any damage, modification, or repair; instrument maintenance logs; and calibration/calibration verification run logs.

I.4.5 Laboratory SOPs. Laboratories shall be required to maintain written, approved laboratory-specific SOPs for all methods and general operations. Laboratory-specific SOPs that fully detail the actual procedures and documentation used to implement performance-based methods are required. Simply referencing a given method or method number is not sufficient. Overall, these SOPs should be based on the guidance published by USEPA (EPA QA/G-6). The SOPs shall be written narrative, stepwise descriptions of laboratory operating procedures. The SOPs shall accurately describe the equipment and the actual procedures used in the laboratory. Copies of the SOPs shall be readily available to the appropriate laboratory personnel. Calculations that are performed external to an instrument or in its automation software shall be documented in the SOPs. The SOPs should also identify an appropriate estimation of uncertainty for all measurements by the designation of appropriate class/grade of equipment within the SOP, or by the number of significant figures recorded based upon the accuracy of the equipment used. The format for SOPs may vary depending upon the kind of activity for which they are prepared. However, at a minimum, the following sections shall be included: Title/Signature/Effective Date page; Scope and Application; Method Summary; Sample Preservation, Containers, Handling, and Storage; Interferences and Potential Problems; Equipment and Apparatus; Reagents and Solutions; Procedures; Calculations; Quality Assurance/Quality Control; Corrective Actions, Data Evaluation; MDL Studies/Sensitivity Assessment; Health and Safety; Sample Disposal; References; and Example Forms. Laboratory SOPs shall be given unique identification (ID) numbers. These SOPs shall be controlled documents that are reviewed annually or updated as necessary whenever procedure/method changes are made and a new version number assigned. Retired SOPs shall be maintained on file by the laboratory in case data quality questions arise later.

I.4.6 Document control procedures. The laboratory shall maintain records documenting all phases of sample handling from sample receipt to final analysis. Accountable documents used by laboratories include, but are not limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, instrument printout, and other documents relating to the sample or sample analysis. The laboratory shall use a document numbering and identification system for all documents/logs. All observations and results recorded by the laboratory shall be recorded on either preprinted laboratory forms or permanently bound laboratory logbooks, or entered into secure computer systems. Observations including noting basis for any manual integrations performed are recommended. Pages in both the bound and unbound logbooks shall be sequentially numbered. Preprinted laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person(s) performing the activity at the time the activity was performed. Permanently bound laboratory logbooks shall be dated and signed by the person performing the activity at

the time the activity was performed. All logbook entries shall be in chronological order. All entries shall be recorded in indelible ink. Unused portions of the logbooks shall be "z'd" out. Corrections to logbooks shall be made by drawing a single line through the error and entering the correct information. Corrections and additions shall be dated and initialed. Computer forms shall contain the name of the laboratory and be dated and signed by the person performing the activity at the time the form is printed. Computer systems must be established to maintain the integrity of the data, i.e., verified to ensure accurate capture, processing, manipulation, recording, and reporting of data, configured to restrict access and provide for appropriate backups and audit trails, etc. The laboratory shall retain on record all original observations, calculations and derived data, calibration records, and a copy of the test report for a minimum of five (5) years, or as specified by project requirements if longer periods are defined. In the event of laboratory closure, all records shall be transferred to the appropriate USACE clients.

I.4.6.1 Standard preparation log. Standard preparation logs should document the preparation of all calibration standards and spiking standards associated with the respective analysis (e.g., the initial calibration, CCV, and initial calibration verification (ICV) standards as well as the MS, LCS, surrogate, and postdigestion spike (PDS) spiking standards). The laboratory shall maintain complete internal documentation for all standards and reagents used that allows traceability back to the original source. At a minimum, the standard preparation logs must clearly specify the following for all standards:

- Sources (e.g., manufacturer and lot number for commercial stock solutions)
- Composition (e.g., initial and final concentration of all target analytes, type and purity of standards)
- Preparation and expiration dates
- Unique ID number of the standard
- Reagents and solvents added to standards (including source and lot numbers)
- Name of preparer

When a standard is prepared via the dilution of a stock solution, the spiking volume and concentration of the stock solution and the final volume and concentration of the diluted standard should be specified and documented accordingly. Manufacturer certificates for commercially purchased stock standards must be maintained. When the laboratory prepares its own stock solutions, calculations and conversion factors should be shown in the standard preparation log (e.g., a general formula or sample calculations).

I.4.6.2 Sample preparation log. Sample preparation logs should document all significant sample preparation activities. All reagents/standards used shall be clearly identified (e.g., with lot numbers) on the appropriate laboratory bench log sheets. The sample preparation logs must include the following information:

- Sample and batch ID numbers
- Matrix
- Preparatory method (method or laboratory SOP ID number)
- Date of sample preparation
- Initial volume or weight of the sample processed
- Final volume of the sample processed (after digestion, extraction, or cleanup)

- Percent moisture (for solid samples)
- Reagents and solvents added to the samples (including source and lot numbers)
- Any pH and preservation checks and adjustments performed
- Spiking standards (ID number of the LCS, and MS spiking solutions, volume added, and the final spike concentration)
- Name of analyst

I.4.6.3 Instrument run log. Instrument run logs shall be maintained for each instrument to enable a complete reconstruction of the analytical run sequence. Run sequence logs must indicate the unique identifier appropriated for the instrument used to generate the data, the date of analysis, and the aliquot volume of the sample analyzed (e.g., the injection volume for chromatographic methods). The time of analysis must be specified for chromatographic methods. The order in which field and QC samples are collected and presented should be consistent with the temporal order in which the analyses were performed. Run logs must clearly indicate which field and batch QC samples are associated with each initial calibration, ICV, and CCV. Instrumental analysis logs are particularly important since they provide the basic link between the sample analyses and QC data. Computer logs may be used if all of the preceding information is captured.

I.4.6.4 Computer/instrument outputs. Computer/instrument printouts or other independent information can be incorporated into logbooks if such printouts can be permanently affixed to the appropriate logbook.

I.4.6.5 Electronic data management. Electronic data management systems shall be verified by the laboratory to ensure accurate data transfer, reduction, and reporting. All aspects of the data management system shall be fully documented as compliant with USEPA Good Automated Laboratory Practices (GALP) requirements (EPA 2185).

I.4.7 Laboratory quality assurance procedures. The laboratory shall ensure the quality of results by maintaining an integrated QA system of activities involving the planning, implementation, assessment, reporting, and quality improvement of data. Refer to ISO/IEC (1990) Guide 25 and American National Standards Institute/American Society for Quality Control (1994) for additional information. These activities are typically performed or facilitated by the Laboratory QA Officer and include the performance of periodic audits (system and technical); participation in proficiency testing programs/interlaboratory comparisons, routine analysis of certified reference materials or second source reference materials, and monitoring method performance (sensitivity, precision and bias) through an evaluation of the MDL study or MDL check sample, and batch QC sample (MB, LCS) control ranges/charts.

I.4.7.1 Laboratory audits. As noted in Section I.4.3.2, annual laboratory audits shall be conducted internally for each analytical area to verify the following at a minimum: procedures are compliant with SOPs; documentation practices are complete and traceable to a certified source(s); data reviews are complete, well-documented, and effective; and data reporting practices, including electronic or manual data transfer and client report generation, are accurate and complete. All audit findings, any corrective actions, root cause determination, etc. shall be fully documented in QA reports to management. The QA officer shall document that all corrective actions necessary are verified complete within a reasonable time frame. Audits performed by external agencies or accrediting authorities shall not substitute for internally conducted laboratory audits.

I.4.7.2 Laboratory method performance monitoring using LCS.

I.4.7.2.1 The laboratory shall generate in-house warning (2-sigma) and control (3-sigma) limits for all target analytes from LCSs, defined in Section I.10.2.2. The LCSs are prepared from interference-free aqueous and solid matrices in order to evaluate the quality of the method performance. These "mean" control limits/charts are generated from bias measurements (e.g., LCS recoveries) to assess the method performance and data quality over an extended period of time. The "warning" and "control" limits for mean control charts set at 2-sigma and 3-sigma approximate the 95 and 99 percent confidence intervals, respectively. A minimum of 30 points should be used to establish these control ranges or charts. In addition, data from all analyses (including method failures) should be used to generate the limits to prevent diminishing the ranges by biasing the data input. Outliers may be excluded from the data if proper QA procedures are employed such as using appropriate statistical tests (e.g., Dixon's Extreme Value test, Discordance test). It would not be necessary to maintain graphical control charts for all target analytes. It is recommended that a representative subset of target analytes for each method be chosen for control chart generation to observe method trends. These control ranges should be updated every 6 months, and reviewed by the QA officer annually at a minimum. Additionally, range control charts may be used to evaluate precision between interbatch LCSs. Range control charts set the 95 and 99 percent confidence intervals at 2.456-sigma and 3.268-sigma for the warning and control limits, respectively. ***Because so many laboratories mistakenly apply the 2-sigma and 3-sigma factors to calculate precision control limits in lieu of the correct factors noted, caution should be exercised when comparing precision control limits between different laboratories.***

I.4.7.2.2 Evaluate laboratory control limits against the measurement quality objectives presented in the project DQOs, the published reference method, or this guidance to survey the need for method evaluation or modifications. Note the baseline measurement quality objectives summarized in Tables I-1 through I-8 are intended for evaluation of batch control acceptance and may not be reflective of a laboratory overall performance as depicted by their internal control limits. Evaluate the calculated mean for a general assessment of the method systematic bias and review of representative control charts for evidence of analytical trends. Information gathered should be used to troubleshoot analytical problems associated with method implementation, with suggestions offered for quality improvements and corrective action to tighten limits.

I.5 Laboratory Sample Handling Requirements

I.5.1 Sample receipt. The receiving laboratory's chain-of-custody, sample storage, and dispersement for analysis shall be documented per specific laboratory SOPs and project requirements. ***Information on project custody, analysis, and data reporting requirements as noted in the SAP and highlighted on the Laboratory Notification Checklist or similar should be received by the laboratory prior to (or accompanying as with the Laboratory Notification Checklist) the first shipment of incoming samples.*** Individual Cooler Receipt Forms or similar shall be used by the laboratory for each cooler to verify sample condition, including proper sample containers, volumes, preservation, etc., and document any problems noted. Corrective action will be required for any deficiencies identified. Refer to Chapter 3, Figures 3-4 and 3-3, for facsimiles of examples of the laboratory notification sheet, and cooler receipt form, respectively. ***It is recommended that all coolers contain at least one temperature blank.*** The temperature blank should be a 40-mL volatile organic analysis vial filled with water and placed in a representative position inside the cooler. Multiple vials could be used, if needed. The laboratory should document when the temperature blank was positioned inappropriately or was not representative of the cooler temperature measurement. Sample log-in procedures shall follow the facsimile cooler receipt form (Figure 3-3). The chain-of-custody form, any shipping documents, completed cooler receipt forms, telephone conversation record forms, and any corrective action forms will be maintained by the laboratory for each shipment and included in the reporting package when the results are submitted.

I.5.2 Sample storage. The laboratory shall provide an adequate, contamination-free, and well-ventilated work space for the receipt of samples. All samples and their associated extracts shall be stored under conditions that will ensure their integrity and preservation and are demonstrated to be free from all potential contaminants. Sufficient refrigerator space shall be provided for the proper storage of all samples and their associated extracts. Samples shall not be stored with standards. Samples designated for volatile organics testing shall be segregated from other samples while samples suspected to contain high levels of volatile organics (e.g., underground storage tank soil samples) should be further isolated from other volatile organics samples. ***In the absence of project-specific criteria, samples and their associated extracts shall be stored for a minimum of 60 days after receipt of the final data report for those samples.*** After that time, the laboratory is responsible for the disposal of the samples and their associated extracts in compliance with all Federal, State, and local regulations unless arrangements have been made for the return of any unused sample portions to the site.

I.5.3 Sample security and tracking. The laboratory shall maintain the integrity of the samples received, their associated extracts, and the data generated. Limited and controlled access to all laboratory areas shall be maintained. ***If required by the project, the laboratory should maintain sample and extract chain-of-custody within the laboratory at all times through the use of appropriate documentation and forms; otherwise strict internal chain-of-custody would not be required.***

I.5.4 Sample holding times. Extraction/digestion holding times shall be defined from the date/time of sample collection in the field to the date/time when the sample is first exposed to the extraction/digestion solvent. Analysis holding times shall be defined from the date/time of sample extraction to the date/time of sample analysis. It is required that laboratories maintain documentation that clearly show the dates (and times when applicable) for all sample handling/manipulation processes. Samples should be analyzed as soon as possible after sample collection. Published holding times are generally considered maximum times that samples may be held before analysis and still be considered compliant with method guidelines. Sufficient time should be allowed for the repreparation or reanalysis of samples within holding times should calibration, method, or quality control failures occur.

I.6 General Analysis Requirements

I.6.1 Project application. The requirements presented in this guidance shall be applied to all analytical methods unless specifically overridden by project-specific requirements. Target analyte lists are variable and are dependent upon project-specific considerations. Examples of common target analyte lists are included for eight SW-846 methods.

I.6.2 Method development/initial demonstration of capability. For each method performed, the laboratory shall maintain documentation that demonstrates each analyst's ability to perform the method within the sensitivity and precision/bias limits as stated in the published method, and any requirements outlined within the project SAP. Repeat these procedures when there is significant change in the method, instrumentation, or personnel. For each new method the laboratory shall perform and maintain documentation for the following:

- Develop a detailed SOP before implementation of that method. Refer to Section I.4.5 for SOP requirements.
- Evaluate method sensitivity by performing an initial MDL study for each matrix per Section I.3.3.7.1. Due to the difficulty in obtaining a solid interference-free matrix for metals determinations, process spiked reagent water for both the aqueous and solid digestion methods

to estimate aqueous and solid MDLs for graphite furnace atomic absorption (GFAA) and ICP analyses.

- Determine an appropriate MQL and MRL for each compound and matrix based upon the calculated MDL and the guidance established in Sections I.3.3.7.2 and I.3.3.7.3.

- Perform an initial demonstration for the method, noting all key employees' (i.e., technicians and analysts) ability to perform the method within the precision/bias limits as stated in the published method. A minimum of four laboratory control samples shall be carried through the method at the same time, or over a period of consecutive days. This control sample shall be obtained from an outside source, if available, or from a lot independent of the calibration standards. The concentration of each target analyte shall be approximately 10 times the MDL. Using the four results, calculate the mean recovery and standard deviation for each parameter or target analyte of interest. Compare the method precision and bias of the laboratory to the method performance summary presented within the published reference method. If any target analytes exceed the acceptance range, the performance is unacceptable. For all unacceptable target analytes or parameters, corrective actions shall be taken to locate the source of the problem, and the test should be repeated. The laboratory must maintain documentation for each analyst performing analysis.

I.6.3 Continuing demonstration of capability. All analysts shall be required to demonstrate their continuing capability to perform any given method by ensuring the following:

- All applicable SOPs are kept current and represent the current implementation of the method by the laboratory.

- The sensitivity of each method is demonstrated quarterly by analyzing the MDL check sample, or annually by an MDL study.

- Any adjustments to the MQL based upon noted changes in method sensitivity are made.

- A minimum of one blind PE sample is analyzed successfully on an annual basis.

- The precision and bias of the method are demonstrated by analyzing laboratory control samples and other QC check samples with each batch of samples processed, and monitored by review of method control ranges/charts.

I.6.4 Data fraud/inappropriate practices. The data produced by a laboratory typically provide the primary basis for environmental cleanup decisions and enforcement actions. The data may also end up in court. ***Data must be produced according to the project-specific requirements as specified in the final approved project documents.*** The laboratory must be aware of these requirements and be able to show that these requirements were met. Documentation that would clearly show how all analytical values were obtained must be maintained. ***The unfortunate aspect of data fraud/inappropriate practices is the inability to readily detect them without significant cost. Project QA procedures employed should be designed to help deter and expose any misrepresentation of data. Refer to Section I.1.2 for information on application of various QA procedures to aid in the prevention of fraudulent activities.***

I.6.4.1 Data fraud. Data fraud can be loosely defined as a gross deviation from contract-specified or method-specified analytical practices, combined with the intent to conceal the deviation. The difference between poor analytical judgment and fraud may be assessed in the documentation of intent within laboratory records. Gross deviations from specified procedures should be investigated for potential fraud, and findings

of fraud should be prosecuted to the fullest extent of the law. The following are examples of fraudulent practices:

- Inappropriate use of manual integrations to meet calibration or method QC criteria. For example, peak shaving or peak enhancement are considered fraudulent activities if performed solely to meet QC requirements.
- Time travel of analyses to meet method 12-hour clock requirements.
- Falsification of results to meet method requirements.
- Reporting of results without analyses to support (e.g., dry-labbing).

I.6.4.2 Inappropriate practices. Several inappropriate practices have also been identified that deserve prudent action. ***Issues of this caliber should not be tolerated, and corrective action should be taken immediately to resolve all matters between the laboratory and the data user.*** These inappropriate practices may include the following:

- Selective exclusion of data to meet QC criteria (i.e., initial calibration points dropped without technical or statistical justification).
- The repetitive analysis of QC samples and the reporting of only the best result to avoid corrective actions. For example if two or more CCVs are analyzed in an automated run sequence. The last CCV passed but the first CCV fails, then it would be inappropriate to report only the second CCV, and to not perform appropriate corrective actions.
- Multiple instrument or method blanks should not be analyzed prior to other QC samples as a means to address carry-over problems, when these blanks are not analyzed before environmental samples also. QC samples must be analyzed in a manner that is representative of the manner in which environmental samples are analyzed, and not given preferential treatment.
- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported, or QC control limits presented within LQMP that are not indicative of historical laboratory performance or used for batch control.
- Notation of matrix interference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (e.g., MB or LCS).

To avoid miscommunication, a laboratory must have an SOP that presents correct procedures for manual integrations. As well as clearly documenting all errors, mistakes, and basis for manual integrations within the case narrative, when manual integrations are necessary. Include corrective actions taken, when necessary, and provide appropriate peer review of this information. ***Notification should also be made to the appropriate people so that appropriate corrective actions can be initiated.*** It is recommended that laboratories also maintain an electronic audit trail that clearly shows all changes to data, who made the change, date, and why.

I.6.5 Analytical standards preparation and traceability. The laboratory shall have, in-house, the appropriate standards for all target analytes. These standards can either be prepared from neat high purity bulk materials or purchased as certified solutions. A critical element in the generation of quality data is the

purity/quality and the traceability of the standard solutions and reagents used in the analytical operations. Primary reference standards and standard solutions used by the laboratory shall be obtained from reliable commercial sources (i.e., National Institute of Standards and Technology (NIST), USEPA, etc.) to ensure the highest purity possible. Certificates shall be available upon request that verify the purity or concentration of each standard. The use of correction factors for all standards that are not at least 99.9 percent pure for inorganics and 96 percent pure for organics will be required. Care should be exercised in the proper storage and handling of all standards and standard solutions. The laboratory shall continuously monitor the purity or quality of reagents and standard solutions through a series of well-documented procedures. Requirements for standards repreparation shall be based on unacceptable performance. For example, initial calibration standards shall be verified with a freshly prepared ICV. For analyses that allow analytical sequence initiation by a CCV, the frequency of standard repreparation will be based on whether standard performance is compliant with the method acceptance criteria. The quality of CCVs failing to meet method criteria should be verified against a freshly prepared CCV. In general, stock and working standards shall be checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. All standards and standard solutions are fully documented to comply with Section I.4.6.2.

I.6.6 Sample screening. It is highly recommended that the laboratory screen samples or extracts by methods of their choice to determine which target analytes are present and at approximately what levels.

I.6.7 Target analyte listings. ***Target analyte lists necessary for a project should be identified within project contract documents based upon project-specific DQOs***. However, for instances where a particular SW-846 method is specified but the target analyte list for the method is not, Tables I-9 through I-14 may be used to identify target analyte lists for the following SW-846 methods: 8021, 8081, 8082, 8260, 8270, and 8330. These lists were compiled of target compounds common to the various versions of each SW-846 method, and as noted in SW-846 Chapter 2. Note, however, that the most recent versions of these references may contain additional target compounds not included here. The organic target analyte lists (Tables I-10, I-11, I-12, and I-13) were augmented to include those compounds included within the Target Compound List as defined by the USEPA CLP. ***Each list should be reviewed based upon project data needs and edited accordingly. Special considerations for target analyte reporting for the following methods should be evaluated and clearly identified within project contract documentation.***

I.6.7.1 Method 8021: VOC by GC/photoionization detector/Hall electrolytic conductivity detector (HECD). The target analyte list for Method 8021 includes those analytes previously associated with deleted SW-846 Methods 8010 and 8020 and some additional target analytes. ***Therefore, depending upon project requirements, the entire 8021 target analyte list or a subset may be specified for the project. The following target analyte lists may apply: the full 8021 target analyte list; HVOs (halogenated volatile compounds) (compound list from deleted Method 8010); AVOs (aromatic volatile compounds) (compound list from deleted Method 8020); or BTEX (benzene, toluene, ethylbenzene, and xylene).***

I.6.7.2 Method 8081: Pesticides by GC/electron capture detector (ECD). ***Note whether multicomponent pesticides (i.e., chlordane and toxaphene) are actually analytes of concern.*** The additional instrument and method QC samples required for these multiple-component analytes significantly increase the level of effort for this method. ***It should also be determined if chlordane quantitation should be performed and reported as technical chlordane or the individual chlordane isomers (i.e., alpha and gamma chlordane).*** In the absence of guidance to the contrary, assume that quantitation is required for toxaphene and the individual chlordane isomers (rather than for technical chlordane). Recently promulgated revisions of Method 8081 do not include PCBs as target analytes. Therefore, guidance on PCB reporting is not included here. Reference Section I.6.7.3 for additional information on reporting considerations for PCBs.

I.6.7.3 Method 8082: PCBs by GC/ECD. ***Regulatory aspects of PCBs are based upon the quantitation as Aroclors. However, when not used for regulatory purposes and depending upon project requirements, the results may be reported as individual PCB congeners, or the values summed over an appropriate chromatographic range and reported as total PCBs. When weathered PCBs are encountered and the data use requires the use of Aroclors, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Peaks within the sample chromatogram not related to PCBs should be subtracted from the total area. Full documentation of this approach must be provided in the case narrative when this option is chosen. Caution should be exercised when using differing quantitation techniques for comparability of project data may be reduced. Studies have shown that concentrations derived from samples quantitated as Aroclors were larger than those determined using the congener method. Due to the potential regulatory aspect and unless otherwise indicated, all samples must be analyzed for the PCB compounds as Aroclors.***

I.6.7.4 Method 8330: Explosives by HPLC. Due to the lack of resolution between 2,4-DNT and 2,6-DNT, and between 2-Am-DNT and 4-Am-DNT, reporting of these compounds may be combined and reported as isomeric pairs.

I.6.8 Analytical methods summary. The guidance introduces two inorganic (6010, 7010/7470/7471) and six organic (8021, 8081, 8082, 8260, 8270, 8330) SW-846 analytical methods. ***The guidance has deliberately omitted method revision numbers from the analytical method designations to enforce its application to any revision of the method in use by USACE. Note also that many of the QA/QC principles and policies included herein apply to methods not directly addressed.*** Technical details on the implementation of the eight methods and default limits for performance-based QC parameters are presented. ***As noted, these acceptance limits are considered a baseline standard, but they may be modified based upon project-specific DQOs. Reference EM 200-1-2 for information on establishing project DQOs, and EM 200-1-6 for a review of CDQM elements available to aid in the design of a project CDQM program. Project-specific contract documents (e.g., scopes of work, guide specifications, etc.) should reference or identify all applicable analytical methods and QC elements necessary for the project to assure correct and accountable execution of the work.*** When this information is not available or adequately defined, then the laboratory shall default to using the latest promulgated revision of the appropriate SW-846 method and application of the QC acceptance limits described herein as the default USACE requirements. The following guidance also outlines general requirements that apply uniformly to all methods by subject heading and any additional parameter or method-specific requirements presented in subsequent sections by chemical parameter, analytical technique, or the individual chromatographic method. For general sample handling procedures used during sample preparation, such as requirements for correct sample homogenizing and laboratory subsampling, refer to the guidance established within Instructions E-4 and E-5, Appendix E.

I.6.8.1 Inorganic analytical methods. The inorganic methods presented focus exclusively on metals analyses. This encompasses inductively coupled argon plasma-emission spectroscopy (ICP), GFAA, and cold vapor-atomic absorption (CVAA) methodologies. Project inorganic method requirements should be clearly identified based on project DQOs. ***Note than when the quantitation limit of a metal (e.g., Sb, Pb, As, Tl, and Se by ICP) is higher than the project-required action level, an alternate analytical method capable of achieving a lower quantitation limit for that metal should be used.*** Baseline inorganic QC requirements are discussed in subsequent sections by the individual method and summarized in Tables I-1 through I-8. Classical (wet chemistry) techniques are not addressed directly within this guidance. However, the field of conventional, nonmetals analysis involves a variety of instrumental and wet chemical techniques. Instruments include spectrophotometers and other analyzers.

I.6.8.1.1 Inorganic preparatory methods. Several preparatory method options may exist for each determinative method and matrix. However, comparability of the data generated from different preparatory procedures is not guaranteed, nor likely. ***Therefore, in order to ensure comparability of data generated throughout the life of a project or between different laboratories, proper preparatory methods must be clearly identified for each chemical parameter/matrix and consistent analytical protocols must be maintained.*** Aqueous liquid samples for ICP may be processed by a hotplate technique following Method 3005 or 3010, or by using a microwave technique following Method 3015. Aqueous liquid samples for GFAA are processed by a hotplate technique following Method 3020, or using a microwave technique following Method 3015. ***When a comparison of dissolved metals and total recoverable metals results is anticipated, recommend that both the field-filtered and nonfiltered water samples be subjected to the proper digestion procedures (preparatory method) prior to analyses. This ensures a matrix matching of the acid concentrations between the samples. If only dissolved metals results are required, the preparatory method is optional, and analysis by direct aspiration is allowed. Under these circumstances and per method requirements, the calibration standards must be changed to matrix match the samples analyzed. The matching of acid concentrations between samples and standards assures similar viscosity and surface tensions, which affect aspiration characteristics and thus may vary the resulting concentrations.*** Solid samples are processed for ICP and GFAA by hotplate following Method 3050, by microwave following Method 3051, or by microwave assisted acid digestion of siliceous and organically-based matrices following Method 3052. Hexavalent chromium digestion should follow Method 3060, and preparatory procedures for the CVAA analysis of mercury are incorporated into the individual analytical methods (7470 for liquids and 7471 for solids). ***Proper preparatory procedures to be employed should be identified within the project contract documents (e.g., SOW, SAP, guide specification, etc.). When the method of digestion is not specified, the laboratory must attempt to obtain this information from appropriate USACE project technical personnel.*** In lieu of project-specific information, the default preparatory procedures shall follow hotplate techniques following Method 3005 for ICP and Method 3020 for GFAA (3005 for antimony) for aqueous matrices, and Method 3050 for solid matrices. It should be noted that future updates of SW-846 are anticipated to combine these preparatory methods under a common methodology.

I.6.8.1.2 Method 6010. This method is used to determine the concentrations of select metals in the digestates of liquid and solid matrices, using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The requirements apply to simultaneous or sequential ICPs. ICPs may be equipped with a torch that is viewed from the radial or axial (e.g., trace ICP) position. For the ICP, mass spectral (MS) detectors are also available.

I.6.8.1.3 Method 7010/7470/7471. The SW-846 7000 series methods are used to determine the concentrations of select metals in the digestates of liquid and solid matrices, employing flame, graphite furnace, gaseous hydride, and cold vapor techniques in conjunction with atomic absorption spectroscopy. This discussion will be limited to GFAA with an appropriate background correction system. Recommend GFAA instruments have a Zeeman background correction capability. ***GFAA is commonly used for several elements due to its sensitivity capabilities.*** It should be noted that the Update IVA of SW-846 includes all GFAA metals methods combined under Method 7010, all direct aspiration (flame) atomic absorption (FLAA) under method 7000; and has deleted all individual metals methods by FLAA, and GFAA. Mercury shall be analyzed by a cold-vapor AA technique following Method 7470 for waters and 7471 for solids. The AA can be integrated with an appropriate cold vapor accessory for mercury analyses, or independent cold vapor units are also available.

I.6.8.2 Organic analytical methods. The principles and QC requirements established within SW-846 Method 8000 apply to all organic chromatographic methods (e.g., GC, GC/MS, and HPLC methods). Therefore, they are presented generally by topic. ***Packed-column methods were formally deleted from SW-***

846 with the promulgation of SW-846 Update III on 13 Jun 1997. These methods, in general, possessed less stringent performance criteria (e.g., column resolution is lower and method QC is less stringent) than their associated capillary column method. ***Due to these factors, the laboratory should default to the use of capillary column methods (e.g., Methods 8260B, 8081A/8082, and 8021B for the deleted Methods 8240, 8080, and 8010/8020, respectively).*** The laboratory shall not use capillary columns in conjunction with packed column methods in order to apply less stringent QC criterion.

I.6.8.2.1 Organic preparatory methods. Several preparatory method options may exist for each determinative method and matrix. However, comparability of the data generated from different preparatory procedures is not guaranteed nor likely. ***Therefore, in order to ensure comparability of data generated throughout the life of a project or between different laboratories, proper preparatory methods must be clearly identified for each chemical parameter/matrix and consistent analytical protocols must be maintained.*** Liquid samples may be prepared for extractable organic analyses using a separatory funnel following Method 3510, a continuous liquid-liquid extractor following Method 3520, or solid-phase extraction by Method 3535. Liquid samples for purgeable organic analyses utilizing purge and trap procedures follow Method 5030. Nonaqueous samples should be prepared by solvent dilution techniques following Method 3580 for extractable organic analyses and Method 3585 for purgeable analyses. Solid samples may be processed for extractable organic analyses by soxhlet extraction procedures following Method 3540, automated soxhlet by Method 3541, pressurized fluid extraction by Method 3545, or ultrasonic extraction procedures by Method 3550. For petroleum hydrocarbons and organochlorine pesticides/ PCBs analyses, a supercritical fluid extraction may be used following Method 3560 and 3562, respectively. ***Typically, Method 3550 (sonication) is used to prepare solid samples known to have high concentrations of target analytes, whereas Method 3540 (soxhlet), 3541 (soxhtet), or 3545 is generally used in an unknown situation or when low-level concentrations are known or suspected.*** Solid samples for purgeable organic analyses utilize Method 5035. ***Several notable changes in the protocols covering soil sampling/analysis preparation have occurred with the promulgation of Method 5035. These changes will require a significant increase in the coordination between field and laboratory personnel. Refer to USACE policy guidance (Headquarters, USACE 1998) for details on implementation. When the method of preparation is not specified, the laboratory must attempt to obtain this information from appropriate USACE project technical personnel.*** If no information is provided for the project-specific preparatory methods required, the default preparatory procedures for extractable organic analyses shall follow Method 3520 for aqueous samples; Method 3540 or 3541 for solid samples; and those noted previously for purgeable organic analyses. ***It is anticipated that project field work will entail the use of proper sample handling protocols that result in the acquisition of a representative sample. These include the use of appropriate sample containers, obtaining sufficient sample volumes, and proper preservation techniques based on the anticipated chemical analyses. Refer to Appendix B for information on proper sample containers, sample volumes, and preservatives necessary. As noted in Section I.5.1 these items are verified upon sample receipt, and any discrepancies notified back through appropriate channels. For chemical parameters that do not allow this assessment during sample login (e.g., VOCs), verification is done post-sample subsampling or analysis, and any problems are noted within the case narrative.*** Whenever possible, a quantitative transfer of the entire (1-liter) aqueous liquid sample is made to ensure no loss of target analytes through the adhesion of contaminants on the walls of the sample bottle. A solvent rinse should be performed to avoid this loss. This procedure, however, may not be possible when significant amounts of sediment are present within the water sample. ***Due to the problems these fines may invoke on the extraction process, recommend that appropriate project technical personnel be contacted to verify the procedures to employ (e.g., decanting water sample, physical separation of the phases and subsequent analysis of each, etc.).***

I.6.8.2.2 Organic cleanup methods. If significant nontarget interference exists, corrective action shall include implementing appropriate cleanup procedures. Dilution techniques should not be used in preference to cleanup procedures for organic methods. The laboratory shall have a minimum capability of at least one cleanup method for each type of organic analyses for which it provides services. Refer to the individual determinative methods and Method 3600 to identify recommended cleanup methods based on the type and concentration of interferences present, the selectivity of the determinative method, and project method reporting limit requirements. However, analyst professional judgment should also be used to identify appropriate cleanup techniques to employ. ***If cleanup procedures are not routinely employed by a laboratory, a formal notification procedure must be in place to advise the client of this.***

I.6.8.2.3 Method 8021. This method is used for the analysis of select VOCs in aqueous and solid matrices by purge and trap device according to methods prescribed previously and subsequently analyzed by GC using an HECD and photoionization detector in series.

I.6.8.2.4 Method 8081. This method is used to determine the concentrations of select organochlorine pesticides in the extracts of liquid and solid matrices, using fused-silica capillary columns with an ECD. ***Method 8081A no longer includes PCBs as target analytes to eliminate the complications inherent in the combined pesticide/PCB analysis. Therefore, PCB analysis must be performed using Method 8082. This may be accomplished by submitting an additional environmental sample for PCB analysis; or the extract may be split prior to implementation of any cleanup procedures, processing individual extract portions for pesticide analysis following Method 8081 and the other portion for PCB analysis following Method 8082.***

I.6.8.2.5 Method 8082. This method is used to determine the concentrations of select PCBs as the seven Aroclors, as individual PCB congeners, or as total PCBs in the extracts of liquid and solid matrices, using fused-silica capillary columns with ECDs. ***Refer to project required chemical parameters and Section I.6.8.2.4 in order to determine the necessity for an additional environmental sample for PCB analysis, or the use of an aliquot from the extract (prior to cleanup procedures) for both pesticide and PCB analyses.***

I.6.8.2.6 Method 8260. This method can be used for the analysis of select volatile organic compounds (most compounds with boiling points below 200 °C) in aqueous and solid matrices by purge and trap device according to methods prescribed previously and subsequently analyzed by GC/MS. Volatile water-soluble compounds can be analyzed with this method but higher quantitation limits may apply. A notable deviation allowed by Method 8260B versus 5030 is the utilization of a heated purge for aqueous samples.

I.6.8.2.7 Method 8270. This method is used to analyze the extracts of aqueous and solid samples for semivolatile organic compounds, also referred to as base/neutral and acid extractables. The extracts are analyzed by GC/MS using a capillary column.

I.6.8.2.8 Method 8330. This method is used for the analysis of select explosives in the extracts of solid and liquid matrices. The extracts are analyzed by HPLC with a UV detector, using C-18 and cyanide reversed-phase columns as the primary and confirmatory columns, respectively. The method specifies extraction procedures for solid samples, and low-level and high-level aqueous samples. In general, aqueous samples for low concentration are extracted by a salting-out extraction procedure using acetonitrile and sodium chloride. Aqueous samples for the high concentration are diluted with acetonitrile, filtered, and analyzed by direct injection. Soil and sediment samples are extracted using acetonitrile in a cooled ultrasonic bath and filtered prior to analysis. ***Project-specific approval should be sought for the use of solid***

phase extraction (Method 3535) in lieu of the low-level salting out procedure described in Method 8330, or the use of a photodiode ray detector as the confirmation technique.

I.7 Preliminary Method Setup

In addition to the general items noted in Section I.6.2, method initiation must include the following procedures as applicable.

I.7.1 Inorganic analyses - Method 6010.

I.7.1.1 Linear dynamic range. The upper limit of the linear dynamic range for each ICP must be determined for each analyte wavelength used in order to determine an appropriate concentration for the high calibration standard. This is done for each analyte by analyzing successively higher standard concentrations (approximately 3 to 5 standards) until--because of curvature--the highest analyte concentration is ± 10 percent of the "expected" concentration obtained by extrapolating the calibration line from the lower standards. The concentration chosen for the highest standard must then be chosen below the upper limit of the linear range. The linear dynamic range must be checked initially and whenever there is a significant change in instrumental hardware or operating conditions. If the ICP is routinely calibrated using one standard and a blank, the linear dynamic range must be checked every 6 months.

I.7.1.2 Interelement spectral correction factors. All interelement spectral correction factors must be determined per method requirements initially and updated at least once every 6 months, based upon failure of the interelement check standard, or whenever there are significant instrument modifications.

I.7.2 Organic analyses - Method 8000 series. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Excessively wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. Retention time windows must be determined as specified in the latest revision of Method 8000 for all chromatographic methods, except when MS or Fourier transformed infrared (spectroscopy) detectors are employed. Calculate absolute retention time windows for each analyte and surrogate for each chromatographic column employed per method instructions. New retention time windows must be established whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The use of reasonable "default" values, programmed into instrument software for the width of the retention time window, is allowed if the laboratory demonstrates that the calculated 3-sigma width is consistently less than the default width, and the default width is not "excessively large" (i.e., more than 1 to 2 percent of the absolute retention time).

I.7.2.1 Method 8081. Retention time windows must be established as specified in Section I.7.2 for each surrogate and single-component pesticide target analyte, and for at least three to five characteristic peaks of multiple-component pesticides. For multicomponent pesticide standards, the analyst should also rely heavily on pattern recognition and the analyst's experience in the interpretation of the chromatograms.

I.7.2.2 Method 8082. Retention time windows will vary based upon the project requirements for PCB quantitation as noted in Section I.6.7.3. Absolute retention times will be used when identification of PCBs as Aroclors is performed. Retention time windows must be established as specified in Section I.7.2 for each surrogate and congeners or for at least 3 to 5 characteristic peaks of each Aroclor. If PCB congeners

are quantitated, normally internal standard calibration techniques are used and relative retention times are determined.

I.8 Instrument Performance Checks

Several methods outline additional QC procedures to verify the instrumentation is in good working condition. These QC samples must be analyzed and meet method-specified acceptable limits prior to commencing sample analyses.

I.8.1 Method 6010 - Interference check standard (ICS). An interference check standard (ICS) must be analyzed at the beginning of the analytical sequence to verify the correction factors established in Section I.7.1.2 are valid. The ICS typically consists of a set of solutions: ICS-A contains only the interferents (at relatively high concentrations) and ICS-AB contains both the interferents and the analytes of interest. The interferents in both solutions must be present at the concentrations that are at least as high as the high-level calibration standard. The ICS-AB solution must contain the analytes of interest (the metals that are not interferents) at concentrations approximately midlevel. The metals of interest in the ICS-AB solution must be within 20 percent of their expected values. When the ICS check is unacceptable, take corrective action to remedy the failure. Check that the background correction factors applied are appropriate, and readjust if necessary. If the ICS fails immediately after the daily initial calibration, recalibrate and reanalyze the ICS. If the ICP can display overcorrections as negative readings, then the ICS-A solution alone may be used to check for interferences. If the analytes of interest are within two times the absolute value of the MDLs (± |MDLs|), the ICS check is acceptable and the ICS-AB solution need not be analyzed.

I.8.2 Method 8081 - Injection port inertness check. Verify injection port inertness by performing percent breakdown checks for 4,4'-DDT and endrin as specified in Method 8081. The midlevel standard containing only endrin and 4,4'-DDT must be analyzed at the beginning of the analytical shift/sequence, before the initial calibration or the continuing calibration verification. If the percent breakdown is not • 15 percent for either DDT or endrin, perform injector maintenance (e.g., column clipping). Do not proceed with the calibration or analysis until the percent breakdown for each compound • 15 percent.

I.8.3 Methods 8260 and 8270 - Mass spectrometer (MS) tuning. Verify that the MS meets standard mass spectral abundance criteria prior to initiation of any analyses by the injection of BFB (4-bromofluorobenzene) tune standard for Method 8260 and DFTPP (decafluorotriphenylphosphine) for Method 8270. The tune standard must be analyzed at the beginning of the analytical shift/sequence and every 12 hours of continuous analysis. The 12-hour clock starts at the time of injection of the tune standard. Recommend evaluating the ion abundance by using any of the following scan scenarios: use one scan at the apex peak, use the mean of the apex and the preceding and following scans or mean of a symmetric pattern of scans about the apex, or use the average across the entire peak. The tune must satisfy the ion abundance acceptance criteria listed within the appropriate method. Background correction should be compliant with method specifications and employed only for the purpose of correcting for instrument background ions. If a 12-hour tune fails, take corrective action (e.g., clean the MS source) and reinject the tune standard (BFB/DFTPP). Do not proceed with analysis until the tune is acceptable.

I.8.4 Method 8270. In order to verify column condition and injection port inertness, the DFTPP tune standard shall contain appropriate volume of 4,4'-DDT, benzidine, and pentachlorophenol as stated within Method 8270.

I.8.4.1 Injection port inertness check. Similar to Method 8081, the injection port inertness of the GC portion of the GC/MS is evaluated by the percent breakdown of 4,4'-DDT. This procedure is done to

verify acceptable instrument performance, regardless of whether DDT is a target analyte. The percent breakdown of 4,4'-DDT to 4,4'-DDE and 4,4'-DDD should not exceed 20 percent, in order to proceed with calibration procedures.

I.8.4.2 Column performance check. The condition of the GC column is evaluated by the tailing of benzidine and pentachlorophenol. Benzidine and pentachlorophenol must be present at their normal responses, with no visible peak tailing, as demonstrated by the peak tailing factors. The calculation of peak tailing factors can be found on Figure 13 of Method 625, 40 CFR 136, App. A. The acceptance criteria for the peak tailing factor for benzidine is <3.0 and for pentachlorophenol is <5.0.

I.9 Calibration Procedures and Frequencies

The calibration of instruments and support equipment is required to ensure that the analytical system is operating correctly and functioning at the proper precision, bias (accuracy), and sensitivity. ***The frequencies of calibration and calibration verification are presented in the following sections, based upon the various analytical methods and industry standards, or may be changed based upon project-specific DQOs.***

Tables I-1 through I-8 highlight key information on calibration procedures and acceptance limits for each SW-846 method discussed.

I.9.1 Analytical support areas calibration verification. Suggest referring to ASTM D 5522 for additional details on the following procedures and performance criteria.

I.9.1.1 Balances. The calibration of analytical balances shall be verified on first daily use at a mass or masses that bracket or are representative of the measurements routinely performed at that balance. The quality of the weights used for this calibration verification shall be documented and shall be in accordance with the quality requirements established within the referenced ASTM standards. Balance calibration verifications shall be documented in appropriate log books. Acceptance criteria shall be clearly identified. Apply a 1 percent performance criterion to top-loading balances, and 0.1 percent to analytical balances. Refer to ASTM D 5522, ASTM E 319, and ASTM E 898 for additional details.

I.9.1.2 Refrigerators/freezers. All refrigerators and freezers shall be monitored for proper temperature by measuring and recording internal temperatures on a daily basis. The calibration of all thermometers used for these measurements shall be verified at least annually against NIST-certified or NIST-traceable thermometers. Electronic thermometers shall be calibrated at least quarterly. Temperatures shall be recorded in appropriate log books. Acceptance ranges shall be clearly identified. Maintain refrigerators to 4 °C ± 2 °C, and freezers to -10° to -20 °C. Refer to ASTM Method E 77 for additional details.

I.9.1.3 Pipets and other volumetric labware. All volumetric devices, glassware, or lab ware shall be initially inspected, and all cracked or damaged items removed from use. The calibration of variable-volume Eppendorf-type pipets shall be verified at the volume of use, or at two volumes that bracket the range of use on the day of use, or at a minimum of weekly. The calibration of all fixed-volume Eppendorf-type pipets shall be verified monthly. In addition, the accuracy of all nonstandard lab ware (K-D tubes, Zymark tubes, plastic cups, centrifuge tubes, etc.) used to measure the initial sample volume or final volume of sample extracts/digestates must be verified. Accuracy must be verified to within 3 percent. If the check reveals error greater than 3 percent, steps should be taken to improve the accuracy of these measurements, or use alternative procedures that meet this requirement. It is also recommended that the calibration of all other volumetric glassware (flasks and pipets) be verified at the time of purchase for each lot of lab ware received.

Each calibration check shall consist of at least three measurements, and the average calculated and recorded in appropriate logbooks. Refer to ASTM E 542 and ASTM E 969 for additional details.

I.9.1.4 Water supply system. The laboratory shall maintain an appropriate water supply system that can furnish high-purity water capable of meeting the needs of the various analytical areas. The performance of MBs provides an indication of the source water suitability for the analysis. However, the water supply system should be monitored on a regular basis (i.e., daily or before use) by conductivity readouts or implementation of general chemistry parameters. Appropriate general chemistry parameters should be based upon the analysis performed at the laboratory. Refer to ASTM D 1193 for additional details.

I.9.1.5 Other analytical support equipment. Other support equipment used to maintain appropriate temperatures as prescribed within the analytical method (i.e., hotplates, water baths, etc.) should be monitored for compliance with the method-specified ranges. Recommend notation of any critical times or temperatures on appropriate bench sheets or laboratory logbooks.

I.9.2 Initial calibration curve. An analytical instrument is said to be calibrated when an instrumental response can be related to the concentration of an analyte. This relationship may be depicted graphically, and referred to as a "calibration curve." Initial calibration curves must be established based upon the requisite number of standards identified within the method for each target analyte (and surrogate for organics). As previously described in Section I.3.3.7.2, the method quantitation limit(s) shall be established by the laboratory at the low standard for each target analyte. All reported concentrations for target analytes shall be within the high and low initial calibration standards. Data generated below the low standard shall be reported as estimated (J-flag) values. Data generated above the high standard shall be diluted into the calibration range and reanalyzed. The frequency requirements for the initial calibration vary among the individual methods and are presented in the following sections. Tables I-1 through I-8 highlight key information on initial calibrations by method also.

I.9.2.1 Inorganic analyses. For metals analyses, an initial calibration must be performed at the beginning of each analytical shift, and when a CCV fails or significant instrument maintenance is performed. Linearity is acceptable only if the linear regression coefficient r is greater than or equal to 0.995. If r is less than 0.995, take corrective action and recalibrate. As previously noted, classical (wet chemistry) techniques are not addressed directly. But while calibration and standardization procedures vary depending on the type of system and analytical methodology, the general principles outlined in these calibration sections apply universally. Analytical systems for wet chemistry techniques shall be calibrated prior to analyses being conducted. The calibration consists of defining the working range by use of a series of standard solutions. A minimum of five to seven standards is typically used. The calibration shall be verified on an ongoing basis (every ten to twenty samples at a minimum and at the end of the analysis sequence) to ensure that the system remains within specifications.

I.9.2.1.1 Method 6010. The term standard may refer to a "mixed" standard solution containing all the metals of interest (when the metals are compatible) or to a set of standard solutions where each standard contains a subset of the compatible metals of interest. The initial calibration must be established following one of the following options:

- Calibration option 1. Perform the initial calibration with a high-level standard and a calibration blank. The concentration of the single standard establishes the linear calibration range, and must fall below the upper linear dynamic range of the instrument (see Section I.7.1.1). To ensure accuracy of concentrations at the MQL, verification at a low-level standard is prepared from the primary source standard and results must be within ±20 percent of its expected value. If the

20 percent criterion cannot be consistently met, then the concentration of the daily low-level CCV standard (and associated quantitation limits) should be increased until compliance is attained.

- Calibration option 2. The ICP-AES may be alternatively calibrated with three standards and a calibration blank. Evaluate linearity as described in Section I.9.2.1. The concentration of the low-level calibration standard must be set no lower than the MQL for each analyte. The concentration of the high-level standard establishes the linear calibration range, and must fall below the upper linear dynamic range of the instrument (see Section I.7.1.1).

All standards and samples analyzed shall have a minimum of three exposures with the mean of each set of exposures used for quantitation. The exposure times should be optimized for instrumental response and analysis time. Evaluate the RSD for high-level and midlevel standards and calibration verification standards to < 5 percent. Take corrective action (e.g., recheck the appropriateness of the exposure time) and recalibrate if the QC criteria are not met.

I.9.2.1.2 Method 7010. An initial calibration for GFAA must be established from at least three standards and a calibration blank. CVAA calibration requirements are similar to the standard AA procedures but with a minimum of 5 points. Evaluate linearity as described in Section I.9.2.1. For GFAA a minimum of duplicate injections shall be performed for all standards and samples to improve precision and help reduce furnace pipetting uncertainty. The RPD between duplicate injections for all standards shall be <10 percent. If unacceptable, reanalyze the standard. If still unacceptable, perform instrument maintenance as needed to correct the problem and recalibrate.

I.9.2.2 Organic analyses.

I.9.2.2.1 The initial calibration curve is established as specified in the individual methods, using a minimum of five standards for all single-component target analytes and surrogates, and at least three standards for multiple-component target analytes (e.g., toxaphene, chlordane, and PCBs). Care should be exercised to avoid using inappropriate practices identified in Section I.6.4. Once verified, an initial calibration is valid until a CCV fails or significant instrument maintenance is performed. The shapes of calibration curves are typically a linear function between the concentration of each target analyte to the instrument response. However, many method target analyte listings have been expanded to include analytes that cannot be optimized without application of models for quadratic or higher order mathematical functions. When these models are employed, additional standards must be analyzed to accurately delineate the relationship as outlined in Method 8000B.

I.9.2.2.2 Linearity may be determined using linear regression analysis for each target analyte by calculating the correlation coefficient r. The resulting line would normally not be forced through the origin or use the origin as a calibration point unless it is demonstrated that the intercept of the regression line is not statistically different from zero at the 95 percent level of confidence. Another term used to describe the goodness of fit of the line is coefficient of determination r^2 (the squared correlation coefficient). Alternatively for chromatographic methods, the average calibration factor (CF) or response factors (RF) may be calculated for each target analyte. Linearity may be evaluated by calculating the percent relative standard deviation (%RSD) of the CFs/RFs from the initial calibration standards for each target analyte. Linearity is presumed if the correlation coefficient r is equal to or greater than 0.995, if the coefficient of determination r^2 is equal to or greater than 0.99, or if the %RSD is less than or equal to 15 or 20 percent (depending on the method specifications). A visual inspection of the calibration curve should also be used as a diagnostic tool when nonlinear behavior is observed to verify if there is a large percentage error in any particular portion of the calibration curve. If the visual inspection indicates problems, or if one of these criteria is not met, then

the laboratory shall evaluate the following items for implementation based on an understanding of the detector response/contaminant concentration relationship:

- Check the instrument operating conditions or the initial calibration standards used and make adjustments to achieve a linear calibration curve.

- Narrow the calibration range using the same number of standards as required by the individual method. In general, the highest standard would be lowered first. The consequences of all actions taken must also be addressed, i.e., reduction of the calibration range, raising of the MQL, etc.

- Evaluate the use of a nonlinear calibration curve, when applicable. When nonlinear calibration models are used, the resultant line should not be forced through the origin and the origin should not be used as a calibration point. No higher than a third-order (cubic) calibration model shall be used. Note that when a nonlinear calibration model is employed, more data points are needed to maintain at least three degrees of freedom. For example, use of a quadratic function requires a six-point initial calibration curve. The resulting coefficient of determination r^2 should be greater than or equal to 0.99 for this to be considered acceptable.

- Use of alternative techniques (e.g., relative standard error (RSE)) outlined in the USEPA Memorandum, "Clarification Regarding Use of SW-846 Methods" (EPA/SW-846).

- Despite implementation of these alternatives, method limitations may exist that make the acceptance criteria unattainable for all target analytes. Therefore, SW-846 has incorporated an allowance to evaluate the mean of the RSD values for all target analytes in the calibration if this average value is less than the method acceptance criterion. To avoid the inclusion of target analytes showing gross method failure, this approach may be utilized as long as the target analytes do not exceed the criteria established for poor performers in the method-specific tables (I-1 through I-8). ***If the averaging option is employed, the laboratory must communicate the following information within the case narrative to the client: summary of all of the target analytes exceeding method acceptance criteria, the individual RSD results for those compounds, and the mean RSD calculated.***

I.9.2.2.3 Method 8021. Apply the principles as stated in Section I.9.2.2 and summarized in Table I-3. Poor performers for Method 8021 are typically associated with the gaseous compounds and those identified with poor purging efficiency in Table I-9. Marginal failure for %RSD for these compounds shall not exceed 40 percent.

I.9.2.2.4 Method 8081. Several single-component pesticides may coelute on certain GC columns. Therefore, it may be necessary to use two calibration mixtures to ensure sufficient separation for quantitation. Choose calibration mixes to minimize the peak overlap. Surrogates may be calibrated from either mix. For each multiple-component pesticide (e.g., toxaphene), analyze a midlevel standard to aid in pattern recognition. Based upon the positive identification of either compound in the samples, calibrate the instrument for that multicomponent pesticide with a minimum of three standards and reanalyze the extract to enable accurate quantitation. Note that if technical chlordane is required, a separate three-point calibration must be performed using technical chlordane standards. Professional judgment should be employed in conjunction with the method instruction to determine the approach used to calculate the appropriate CF(s) (e.g., the use of total area or selection of a minimum of four to six characteristic peaks for toxaphene and three to five for chlordane). CFs are then used to calculate the mean CFs, standard deviation, and RSD and apply

the principles as stated in Section I.9.2.2 for both single- and multi-component pesticides and as summarized in Table I-4. Marginal failure for %RSD for poor performing compounds shall not exceed 40 percent.

I.9.2.2.5 Method 8082. ***Procedures for initial calibrations will vary based on the project requirements for PCB quantitation as noted in Section I.6.7.3 (e.g., PCBs as Aroclors, PCB congeners, or total PCBs).*** When PCBs are to be determined as Aroclors, external standard calibration techniques should be used; when determined as PCB congeners, an internal standard calibration should be used. Table I-5 summarizes appropriate QC limits.

- Aroclors. The approach taken for an initial calibration will differ depending on the project DQOs. For instance, for projects with a few specific Aroclors associated with the site, recommend the following procedures. Perform the initial calibration using five standards for each Aroclor identified by the project. When samples contain a known mixture of different Aroclors, the analyst may perform a five-point calibration using that Aroclor mixture. When a multipoint calibration is performed for individual Aroclors, calculate and use the calibration factors from a minimum of three to five peaks for those standards and evaluate linearity as presented in Section I.9.2.2. If the PCBs are unknown or the types of PCBs have not been determined, recommend the following procedures. Perform the initial calibration using five standards for a mixture of Aroclor 1016 and Aroclor 1260 standards in order to determine linearity of the detector response. For the remaining five Aroclors, a midlevel standard is analyzed to aid in pattern recognition. Based upon the positive identification of any PCBs in samples corresponding to the Aroclors with only the midlevel standard analyzed, calibrate the instrument for that PCB with a minimum of three standards and reanalyze the extract to enable accurate quantitation. Again, using a minimum of three to five peaks, calculate appropriate CFs for the 1016/1260 and any positively identified PCB standards and apply the principles as outlined in Section I.9.2.2 to evaluate linearity.

- PCB congeners. Table I-11 identifies 19 congeners that have been successfully tested by Method 8082. However, the procedure may be appropriate for additional congeners. When PCB congeners are to be determined, decachlorobiphenyl is recommended for use as the internal standard. Perform a five-point initial calibration using standards containing all PCB congeners. Calculate the RF for each congener in the calibration standards, and evaluate the linearity of the initial calibration using principles as outlined in Section I.9.2.2.

I.9.2.2.6 Method 8260. Apply the principles as stated in Section I.9.2.2, in addition to the following items. Poor performers for Method 8260 are typically associated with the gaseous compounds and those identified with poor purging efficiency in Table I-12. Marginal failure for %RSD for these compounds shall not exceed 30 percent. QC elements and acceptance limits are summarized in Table I-6.

- Verify that the mean RFs for the system performance check compounds (SPCCs) satisfy the minimum RFs requirements specified in Method 8260. If these criteria are not met, evaluate the system (e.g., for standard mix degradation, injection port inlet contamination, contamination at the front end of the analytical column and active sites in the column or chromatographic system). Take corrective action and recalibrate for all target analytes.

- If the regression coefficient $r < 0.965$ or RSD > 30 percent for calibration check compounds (CCCs), this is indicative of system leak or column degradation. Take appropriate corrective action (e.g., instrument maintenance) and recalibrate for all target analytes and surrogates.

I.9.2.2.7 Method 8270. Apply the principles as stated in Section I.9.2.2, in addition to the following items. Poor performers for Method 8270 are typically associated with the compounds that exhibit poor chromatographic behavior. Marginal failure for %RSD for these compounds shall not exceed 40 percent. QC elements and acceptance limits are summarized in Table I-7.

- Verify that the mean RFs for the SPCCs satisfy the minimum RFs requirements specified in Method 8270. If these criteria are not met, evaluate the system (e.g., for standard mix degradation, injection port inlet contamination, contamination at the front end of the analytical column and active sites in the column or chromatographic system). Take corrective action and recalibrate for all target analytes.

- If the regression coefficient $r < 0.965$ or RSD > 30 percent for CCCs, this is indicative of system leak or column degradation. Take appropriate corrective action (e.g., instrument maintenance) and recalibrate for all target analytes and surrogates.

I.9.2.2.8 Method 8330. Perform the initial calibration as specified in Section I.9.2.2 with the following points considered. Marginal failure for %RSD for these compounds shall not exceed 30 percent. QC elements and acceptance limits for Method 8330 are summarized in Table I-8.

- Due to the lack of resolution between 2,4-DNT and 2,6-DNT, and between 2-Am-DNT and 4-Am-DNT, calibrations of these compounds may be based on "isomeric pairs." Improved resolution may be obtained using a Supelco C-18 column with an eluent of 55/45 (v/v) methanol/water at 0.8 mL/min.

- The C-18 column may be substituted with a C-8 column (as the primary column) if 2-NT and 4-NT are not target analytes or project-specific approval is obtained. (These two analytes generally coelute on C-8 columns.) Note that a C-8 column must not be used in place of the confirmatory CN-column.

I.9.3 Initial calibration verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This ICV involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The percent recovery of each target analyte in the ICV is determined from the initial calibration and compared with the specifications for the CCV in each method (except for mercury by CVAA) as outlined in Tables I-1 through I-8.

I.9.3.1 Method 8081. A separate ICV standard is required for each multiple-component target analyte (e.g., toxaphene and chlordane) if a calibration is performed based upon its presence in samples.

I.9.3.2 Method 8082. The ICV standards may be limited to contain a mixture of Aroclors 1016 and 1260 or the project-specified Aroclors.

I.9.4 Initial calibration blanks (ICBs) and continuing calibration blanks (CCBs). ICBs and CCBs are required for inorganic metals analyses to verify the system is free of contamination. The frequency of ICB/CCB analyses is presented in Tables I-1 and I-2 as prescribed within SW-846 Methods 6010 and 7010/7470/7471. The concentrations of each target analyte in the ICB/CCB must be less than or equal to the MDL as presented in Tables I-1 and I-2. Samples must not be analyzed until the ICB is acceptable, and all results must be bracketed by passing CCBs to be considered valid.

I.9.5 Continuing calibration verification (CCV). CCVs are analyzed to determine whether the analytical system is working properly, and if a new initial calibration (and the reanalysis of sample extracts) is required. Calibration verification differs in concept and practice from continuing calibration. In this latter technique, a standard is analyzed and new response factors are calculated, or a new calibration curve is drawn from the analysis of the continuing calibration standard. The former verifies compliance with the initial calibration curve, but does not overwrite the response factors used for the quantitation, nor allows resloping of the calibration curve. Calibration verification shall be used for all analytical methods, calculating a percent drift when the initial calibration is based on regression analysis, and a percent difference when the initial calibration is determined based upon %RSD values. CCV typically involves the analysis of a single primary source standard in the middle of the calibration range, between the concentrations of low-level and midlevel calibration standards. The frequencies of the CCV vary between methods but are related to the type of detector used and sample matrices analyzed. The analysis of more frequent CCVs is recommended for very sensitive detectors and when analyzing difficult matrices. This frequency is typically presented within SW-846 methods as at the beginning of the analytical shift/sequence; every 12 hours of analyses or every 10 to 20 samples; and may include at the end of the analytical sequence. Refer to Tables I-1 through I-8 for details on requirements for CCV implementation and acceptance limits for the individual methods. If these QC criteria are not met, take corrective action to inspect the analytical system to determine the cause and perform instrument maintenance to correct the problem before analyzing a second CCV. If the second CCV is acceptable after system maintenance is performed, recalibration is not required but all sample extracts analyzed after the last acceptable CCV must be reanalyzed. If however, the second CCV fails, a new initial calibration must be performed and all associated sample extracts reanalyzed.

I.9.5.1 Inorganic analyses. A calibration verification pair of a CCB and CCV must be analyzed after every 10 samples (including batch QC samples) and at the end of the analytical sequence as outlined in Sections I.9.4 and I.9.5. Refer to Tables I-1 and I-2 or a summary of CCV implementation and QC requirements.

I.9.5.2 Organic analyses. Calibration verification must be analyzed as outlined in Section I.9.5 and as summarized in Tables I-3 through I-8, in addition to the following:

- For certain organic analyses, additional CCVs at low- and high-level concentrations are recommended, due to the instability of their detectors (e.g., HECD, ECD). Measurement quality objectives (acceptance limits) for the high-level CCV should be in accordance with the midlevel CCV criteria. ***This criterion, however, may not be achievable for the low-level CCV. Therefore, no measurement quality objectives for low-level CCVs are included at this time, and should be identified within project documents based upon the use of the data. For instance, if low-level detection is critical based on project action levels or decision levels, appropriate measurement quality objectives should be determined based on an acceptable level of error to support the use of the data.***

- For methods that contain multicomponent target analytes (e.g., PCBs), typically only a subset of these analytes would be used in the CCV.

- For GC/HPLC methods, concepts similar to that presented for initial calibrations apply. However, methods may possess limitations for certain target analytes that make the stated method acceptance criteria unattainable. Therefore, SW-846 has incorporated an allowance to evaluate the mean of the percent difference (%D) or percent drift values for all reported target analytes in the calibration verification standard to verify whether it is less than the method acceptance criteria. To avoid the inclusion of target analytes showing gross method failure, this approach may be utilized as long as

the target analytes do not exceed the criteria established for poor performers in the method-specific tables (I-3 through I-8). ***In addition, the laboratory must communicate this information within the case narrative to the client. Provide a summary of all of the target analytes exceeding method acceptance criteria, the individual %D values for those compounds, and the mean %D calculated.***

- For GC/HPLC methods, compare the retention time of each analyte in the CCV with the absolute retention time windows established in Section I.7.2. Each analyte must fall within its respective retention time window. If this criterion is not met, the chromatographic system must be adjusted to allow another CCV to meet the criterion, or a new initial calibration performed and new retention time windows established.

I.9.5.2.1 Method 8021. Due to the instability and potential drift of the electrolytic conductivity detector (HECD), the following procedures are highly recommended. When analysis includes the HVO target analytes, suggest alternating the midlevel CCV with high- and low-level CCVs as noted in Section I.9.5.2.

I.9.5.2.2 Method 8081. Due to the instability and potential drift of the ECD, the following procedures are highly recommended. Suggest alternating the midlevel CCV with high- and low-level CCVs as noted in Section I.9.5.2, and also recommend incorporating periodic multi-component pesticide CCVs (i.e., toxaphene and chlordane), when applicable.

I.9.5.2.3 Method 8082. When quantitating for PCBs as Aroclors, a midlevel CCV standard containing a mixture of Aroclors 1016 and 1260 (or Aroclors of interest) must be analyzed. When quantitating for individual PCB congeners, the CCV standard must contain all congener target analytes. Due to the instability and potential drift of the ECD, the following procedures are also highly recommended. Suggest alternating the midlevel CCV with high- and low-level CCVs as noted in Section I.9.5.2.

I.9.5.2.4 Methods 8260 and 8270. Apply the principles as stated in Section I.9.5.2, in addition to the following items. It is further recommended that a CCV be analyzed at the end of the analytical sequence.

- Evaluate the RFs of the SPCCs in the CCV. If the SPCCs do not satisfy the minimum response factor requirements specified by Method 8260/8270, take corrective action and reinject the CCV. However, if CCV remains unacceptable, a new initial calibration must be performed.

- Evaluate the responses and retention times of the internal standards in the CCV as soon as possible. If the retention time for any internal standard changes by more than 30 seconds, or the extracted ion current profile area changes by a factor of two (-50 percent to +100 percent) from that of the midpoint standard of a current initial calibration, inspect the mass spectrometer for malfunctions and take corrective action. Reanalyze any affected samples if required.

- Evaluate the concentration of each target analyte and surrogate in the CCV. Verify that the percent drift or percent difference for the CCCs and all project-specified contaminants of concern are within ± 20 percent of their expected values. Evaluate remaining target analytes to assess instrument stability and survey the need for performing instrument maintenance.

I.10 Laboratory Quality Control Procedures

Laboratory overall method performance shall be monitored by the inclusion of various internal quality control checks that allow an evaluation of method control (batch QC), and the effect of the sample matrix on the data being generated (matrix-specific QC). Batch QC is based on the analysis of a laboratory control sample to generate accuracy (precision and bias) data and MB data to assess the potential for cross-contamination. Matrix-specific QC shall be based on the use of an actual environmental sample for precision and bias determinations from the analysis of MSs, MS duplicates, matrix duplicates, and surrogate spikes, etc. Site-specific PE samples could also be used, if available. The overall quality objectives are to implement procedures for laboratory analysis and reporting of data that are indicative of the degree of quality consistent with their intended use. ***Measurement quality objectives given as QC sample acceptance limits and ranges may be default values established within this guidance, or may be based upon project DQOs.*** Laboratory-generated control ranges are also used for an internal evaluation of method performance and control. ***Deviations from any of these target ranges will result in the implementation of appropriate corrective measures and an assessment of the impact on the usability of the data in the decision-making process.***

I.10.1 Sample batching. The basic unit for application of laboratory quality control is the batch. Samples shall be prepared, analyzed, and reported in batches and be traceable to their respective batches. Batch sizes are normally limited to 20 field samples of a similar matrix but can exceed this by incorporating additional QC samples. Each batch shall be uniquely identified within the laboratory. Samples prepared together would normally be analyzed together on a single instrument. Samples taken from the same site would normally be grouped together for batching purposes within the constraints imposed by the method holding times. However, laboratories may find it necessary to group multiple clients' samples into a single batch. Under these circumstances, additional batch QC samples may be needed that evaluate the effect of the matrix from each site on method performance. Field QC samples, i.e., trip blanks, rinsates, etc., shall not knowingly be used for batch QC purposes.

I.10.1.1 Preparation batch. The preparation batch shall be defined as samples of the same or similar matrix that are prepared together by the same person or group of people within the same time period or within limited continuous time periods, following the same method, using the same type of equipment and same lots of reagents. The laboratory shall have sufficient quantities of extraction/digestion lab ware to meet these requirements. Each preparation batch shall contain the requisite number and type of calibration solutions, blanks, QC samples, and regular analytical samples as defined by the analytical method. These requirements shall be completely defined in the laboratory SOPs and are summarized in part in the following sections. The use of cleanup methods would be included as part of the preparation batch. All field and batch-specific QC samples within the batch should be subjected to all preparatory and cleanup procedures employed.

I.10.1.2 Analysis batch (sequence).

I.10.1.2.1 The analysis batch or sequence or instrument run sequence shall be defined as samples that are analyzed together within the same time period or in continuous time periods on one instrument under the control of one continuing calibration verification. Analysis sequences are bracketed by the appropriate continuing calibration verification standards and other QC samples as defined by the analytical method. In general, if an instrument is not used for periods of time or shut down (e.g., overnight, etc.), then a new analysis sequence shall be initiated. Each analysis sequence shall contain the requisite number and type of calibration solutions, QC samples, and regular analytical samples as defined by the analytical method. These

requirements shall be completely defined in the laboratories' SOPs and are summarized in part in the following sections.

I.10.1.2.2 For samples that are purged and then analyzed immediately, the preparation batch and analysis sequences are combined. For this situation, the batch would normally be defined by the loading of samples into the various purge tubes. This definition has been interpreted differently however. For instance, the loading of purge tubes may be performed all at one time, or may continue throughout the day. In order to ensure ambient environmental conditions throughout the potential loading process, USACE requires a minimum of an MB run every 4 hours, or twice a day when samples are loaded throughout the day.

I.10.2 Preparation batch QC samples. A summary of the minimum required QC samples for each preparation batch follows. All calibrations and QC samples analyzed shall be uniquely identified and traceable to that unique sample preparation batch. Additional QC samples may be required for other batch types based upon project DQOs.

I.10.2.1 Method blank (MB). MBs are analyzed to assess background interference or contamination that exists in the analytical system that might lead to the reporting of elevated concentration levels or false positive data. The MB is defined as an interference-free blank matrix similar to the sample matrix to which all reagents are added in the same volumes or proportions as used in sample preparation and carried through the complete sample preparation, cleanup, and determinative procedures. For aqueous analyses, analyte-free reagent water would typically be used. For soil analyses, a purified solid matrix (e.g., sand) would typically be used, except for metals analyses. The results of the MB analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. Refer to Section I.11.4.1 for measurement quality objectives/corrective action scenarios for the MB. Sample results shall not be corrected for blank contamination.

I.10.2.2 Laboratory control sample (LCS). The LCS is analyzed to assess general method performance based on the ability of the laboratory to successfully recover the target analytes from a control matrix. The LCS is similar in composition to the MB. Aqueous analyses use analyte-free reagent water. For soil analyses, a purified solid matrix (e.g., Ottawa sand, sodium sulfate, or other purified solid) would typically be used. However, due to the difficulty in obtaining a solid matrix that is metals-free, analyte-free reagent water is taken through the appropriate digestion procedures for metals analyses. The LCS is spiked with all single-component target analytes before it is carried through the preparation, cleanup, and determinative procedures. ***A subset of the single-component target analytes containing the specific analytes of interest can be substituted for the full list of target analytes if specified in project-specific contracts or work plans. When multicomponent target analytes are reported, a separate LCS may be necessary if specified by project documents. For Method 8082, the LCS must be spiked with at least one PCB (e.g., 1016/1260 mixture), any project-specified PCBs, or all congeners to support the LCS evaluation.*** The use of solid standard reference materials as the LCS is discouraged for they do not typically include all target analytes, and the acceptance limits associated with them are wide due to the heterogeneity of the spiked matrix. Suggest instead the use of an interference-free matrix (e.g., purified solid or sodium sulfate). When samples are not subjected to a separate preparatory procedure (i.e., purge and trap VOC analyses, or aqueous Hg analysis), the CCV may be used as the LCS, provided the CCV acceptance limits are used for evaluation. ***The spiking levels for the LCS would normally be set at the project-specific action limits assuming that the low standard used for the initial calibration was below this limit. If the low standard used was at this limit or if the site action levels were unknown, then the spiking levels would be set between the low- and mid-level standards.*** The results of the LCS are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. Refer to Section I.11.4.2 for measurement quality objectives/corrective action scenarios for the LCS. The

laboratory shall also maintain control charts or tables for these samples to monitor the precision and bias for the method as outlined in Section I.4.7.2. The precision may be evaluated by comparing the results of the LCS from batch to batch, or by duplicate LCSs. Duplicate LCSs within the same batch are not required, but recommended by the USACE.

I.10.2.3 Matrix spikes (MS). The MS is used to assess the performance of the method as applied to a particular project matrix. A MS is an environmental sample to which known concentrations of certain target analytes have been added before sample manipulation from the preparation, cleanup, and determinative procedures have been implemented. ***Reference project-specific documents for the contaminants of concern, guidance presented in the following sections, or the preparatory and determinative methods to determine target analytes to include within the MS spiking solution.*** If no information is available, include all target analytes within the MS. The spike concentrations of the target analytes would normally be set at the same level as the LCS. ***If target analytes are known to be present in samples from a given site, then the spiking level should be adjusted to a concentration that is approximately two to four times the concentrations of the original target analytes.*** For solid samples, care should be taken to ensure that the original field sample is properly divided into homogeneous fractions when allowed by the method. ***Aqueous samples require the submittal of an additional sample for several chemical parameters, especially organic analyses. Therefore, the sample to be used for the MS should be based on project-specific DQOs and specified in the field to ensure that sufficient sample is available to perform the test.*** From the laboratory perspective, preparation batches require MS frequency at one per preparation batch. The merging of these MS frequencies is often difficult for the laboratory to implement. For instance, batches consisting of samples from multiple sites may require additional MSs to meet project requirements of evaluating the samples within the batch because an MS from one site cannot be used to evaluate the matrix effects on samples from other sites. ***Projects must consider the method(s) employed, previous knowledge of the matrix, and other matrix-specific QC samples to help decide an appropriate frequency for MSs for a given project. As a consequence, an MS may not be included with each shipment of samples submitted to the laboratory. Communication between project and laboratory personnel is essential.*** The results of the MS are evaluated in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Refer to Section I.11.4.3 for measurement quality objectives/corrective action scenarios for the MS. ***When critical decisions are based on the MS sample recoveries, control charts could be maintained for these samples to monitor the bias of the method for each particular matrix***. Sample results shall not be corrected for MS QC excursions.

I.10.2.3.1 Method 6010. ***Unless superseded by project DQOs, it is not necessary to perform matrix spikes for Na, K, Ca, and Mg for aqueous samples or Na, K, Ca, Mg, Fe, Mn, and Al for soil samples.*** The native concentrations of these low-toxicity metals are usually relatively high.

I.10.2.3.2 Method 8081. The MS should be prepared for all single-component pesticides. ***Multicomponent pesticides need not be included within the MS, unless required by project DQOs.***

I.10.2.4 Matrix duplicates (MD) or matrix spike duplicates (MSD). The MD or MSD is used to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. An MSD is a duplicate of the MS as previously described. An MD is an environmental sample that is either divided into two separate aliquots by the laboratory, or requires the submittal of an additional sample. When applicable, care should be taken to ensure that the sample is properly divided into homogeneous fractions. Both the MD and MSD are carried through the complete sample preparation, cleanup, and determinative procedures. ***The requirements for the frequency of MDs or MSDs would normally be specified in the project-specific DQOs.*** The normal use of these QC samples would follow the same requirements as described for the MS. ***In the absence of project-specific DQOs, an***

MD would normally be included with each preparation batch of samples processed where target analytes were expected to be present (e.g., inorganic methods). An MSD would normally be included with each preparation batch of samples processed where target analytes were not expected to be present (e.g., organic methods). The results of the MD or MSD are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the precision of the analysis. Refer to Section I.11.4.4 for measurement quality objectives/corrective action scenarios for the MD or MSD. Control charts can be maintained for these samples to monitor the precision of the method for each particular matrix if required by the project.

I.10.2.5 Surrogates.

I.10.2.5.1 Surrogates are analyzed to assess the ability of the method to successfully recover these specific nontarget analytes from an actual matrix. Surrogates are organic compounds that are similar to the analytes of interest in chemical behavior but are not normally found in environmental samples. Surrogates to be used are identified within the determinative methods. Other compounds may be chosen and used as surrogates, depending on the analysis requirements, whether they are representative of the compounds being analyzed, and whether they cover the chromatographic range of interest. These compounds should be spiked into all samples and accompanying QC samples requiring GC, liquid chromatography, or GC/MS analysis prior to any sample manipulation. As a result, the surrogates are used in much the same way that MSs are used, but cannot replace the function of the MS. The results of the surrogates are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the individual sample determinations. Refer to Section I.11.4.5 for measurement quality objectives/corrective action scenarios for surrogates. Control charts or tables shall be maintained for surrogates contained within the LCS or MB to monitor the accuracy of the method for each particular matrix. Sample results shall not be corrected for surrogate excursions.

I.10.2.5.2 Explosives analysis by Method 8330 is an exception, in that the surrogate used is actually a target analyte. Care should be exercised by the laboratory with the choice of surrogate used, for the potential remains for coelution with target analytes present within the samples. If 3,4-DNT is used as the surrogate, it must not coelute with TNT. If it is not possible to obtain adequate resolution between 3,4-DNT and TNT, another surrogate should be chosen (e.g., 1,2-DNB).

I.10.2.6 Standard reference materials. The laboratory is encouraged to analyze additional natural matrix standard reference materials and participate in external PE programs.

I.10.3 Analysis sequence QC samples. Certain inorganic analyses (metals by ICP and GFAA) incorporate the following additional QC samples to assess method performance without the influence of the preparatory procedures.

I.10.3.1 Post digestion spikes (PDS). PDSs are performed on every sample as a recovery test for Method 7010, and one per batch (one the sample chosen for MS) for Method 6010. However, duplicate injections of each environmental sample may be avoided when the PDS is performed for each sample for ICP analysis following Method 6010. PDSs are prepared by the addition of the primary source standard to an aliquot of the digestate for the same metals and at approximately the same concentration as is used for the MS - i.e., between the low and mid-level standards. Refer to section I.11.4.6 for measurement quality objectives/corrective action scenarios for PDSs.

I.10.3.2 Serial dilutions (SD). A 5X (1:4) SD test may be performed for an analyte to evaluate matrix interference if the analyte concentration in the original (undiluted) sample is at least 50 times the

MDL. SD-matrix effects are suspected if the RPD between the undiluted and diluted result is greater than 10 percent. If this criterion is not met, further confirmation of the interference via implementation of PDS is necessary when matrix interference is suspected, and the calculation of the result through the use of method of standard additions when matrix interference is suspected/confirmed.

I.10.3.3 When SDs are used to address matrix interference, only "best" diluted results (i.e., the lowest dilution that yielded acceptable results) need be reported. However, the reported result must be qualified (i.e., D-flag) and the dilution factor specified. The associated MQLs or MRLs must also be adjusted based on the dilution factor.

I.11 Measurement quality objectives and Corrective Actions

When errors, deficiencies, or out-of-control situations exist, the laboratory's QA program shall include a system of QC activities that measure the system performance to verify that it meets stated requirements and objectives. When the analytical system performance does not meet defined standards, the laboratory shall employ systematic procedures, called corrective actions, to resolve problems and restore proper functioning to the analytical system(s). Laboratory personnel are alerted that corrective actions are necessary under the following conditions:

- QC data are outside the measurement quality objectives for precision and bias.
- Blanks or laboratory control samples contain contaminants above acceptable levels.
- Undesirable trends are detected in spike recoveries or RPD between duplicates.
- There are unusual changes in method detection limits.
- Deficiencies are detected by the QA department during internal or external audits or from the results of PE samples.
- Inquiries concerning data quality are received from a project manager.

Corrective actions are often handled at the bench level by the analyst, who reviews the sample preparation procedures for possible errors and checks the instrument calibration, spike, calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager, or QA department for further investigation. ***Poor performance by the laboratory may result in payment penalties or work being repeated at the contractor's expense. Once resolved, full documentation of the corrective action procedure shall be filed with the project-specific records.*** The following identifies measurement quality objectives and the corrective actions necessary. When qualification of data is necessary (e.g., flagging), refer to Section I.13.3 for details on flagging conventions. The following shall be required in the absence of project-specific requirements:

I.11.1 Incoming samples. Problems noted during sample receipt shall be documented on an appropriate form (the "Cooler Receipt Form"). ***The project manager or appropriate technical personnel shall be contacted immediately for problem resolution.***

I.11.2 Sample holding times. ***If samples cannot be prepared or analyzed within the method-required holding times, the project manager or appropriate technical personnel shall be immediately notified so that an appropriate corrective action plan can be generated. If holding times are exceeded and***

results reported, the resulting data shall be flagged, and a discussion of the impact included within the case narrative.

I.11.3 Instrument calibration. Sample analysis shall not be allowed until all initial calibrations, initial calibration verifications, and instrument blanks meet the appropriate requirements. All CCVs that do not meet method requirements shall result in a review of the calibration, rerun of the appropriate calibration standard for the failed analytes, and, if necessary, reanalysis of all samples affected back to the previous acceptable CCV check for the target analytes that failed. Continued failure of the CCV shall result in the construction of a new initial calibration curve followed by the reanalysis of all samples affected. ***If results are reported when a calibration criterion has been exceeded, then all results reported shall be flagged, and a discussion of the impact included within the case narrative.*** Instrument blanks should be implemented as outlined in the prescribed method.

I.11.4 Method QC samples. Each preparatory batch and analysis sequence must include the appropriate batch and matrix-specific QC samples and standards: i.e., MBs, LCSs, MSs, MDs, MSDs, surrogate spikes, and other method-specified QC. ***All QC shall meet the appropriate project-specific measurement quality objectives and associated corrective actions.*** In the absence of such criteria or actions, the corrective actions as described in the following sections shall be required. Failure of method QC shall result in the review of all affected data. If no errors can be noted, the affected sample(s) may need to be reanalyzed or reprepared and reanalyzed within method holding times, if possible. ***All repreparation and reanalysis necessary due to method failure shall be performed at no cost to the Government. If the situation is not corrected and results reported, then the corresponding data shall be flagged and a discussion of the impact included within the case narrative. The project manager or appropriate technical personnel shall be notified as soon as possible to discuss possible corrective actions should unusually difficult sample matrices be encountered.***

I.11.4.1 Method blanks (MBs). These criteria shall be used to evaluate the acceptability of the MB data if project DQOs do not specify otherwise. The concentration of all target analytes shall be below one-half of the reporting limit (MRL) for each target analyte, or less than 5 percent of the regulatory limit associated with that analyte, or less than 5 percent of the sample result for the same analyte, whichever is greater for the MB to be acceptable. When this criterion is exceeded, corrective action should be taken to find/reduce/eliminate the source of this contamination in the MB. However, sample corrective action may be limited to qualification for blank contamination (i.e., B-flag). When the concentrations of any target analytes within the MB are above one-half the MRL for the majority of target analytes or above MRL for target analytes known to be common laboratory contaminants, assess the effect this may have had on the samples. If an analyte is found only in the MB, but not in any batch samples, no further corrective action may be necessary. Steps shall be taken to find/reduce/eliminate the source of this contamination in the MB. The case narrative should also discuss the situation. If an analyte is found in the MB and in some, or all, of the other batch samples, additional corrective action is required to reanalyze the MB, and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch should be reprepared and reanalyzed with a new MB and batch-specific QC samples. Sporadic cases of contamination may be difficult to control; however, daily contamination would not be acceptable.

I.11.4.2 Laboratory control samples (LCSs). ***The LCS is evaluated by comparing the percent recovery for all of the target analytes to the recovery measurement quality objectives as determined by project-specific DQOs, or the default ranges established in this guidance.*** If target analytes are outside the acceptance windows, corrective action is required. Project DQOs will dictate the corrective actions necessary. Initially, the effect the QC failure has on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it. The case narrative shall

discuss the corrective action taken and any other information. Typically, the LCS would be reanalyzed for the failed analytes only. If the second analysis fails, then the LCS, MB, and all associated samples of the batch would be reprepared and reanalyzed for the failed analytes only. ***If sufficient sample is not available for repreparation and reanalysis or if the corrective action is ineffective, the sample results reported within that batch shall be flagged accordingly, and a discussion of the impact included within the case narrative.*** When multiple (>5) target analytes are reported, the acceptance criteria may allow for the sporadic marginal failure of a few target analytes included within the LCS without requiring reanalysis of the entire batch. ***For methods that report several (>5) target analytes, a small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger reextraction and analysis of the entire batch). The number of target analytes reported for the method will dictate the number of allowable QC failures as given in table I-15.*** Refer to the individual method tables (Tables I-1 through I-8) for details of this concept as it pertains to each of the methods discussed. The marginal failure allowance entails the application of an expanded acceptance criterion.

I.11.4.3 Matrix spike (MS) samples. ***The MS is evaluated by comparing the recovery for target analytes to the recovery windows established within project documents, or those established in Tables I-1 through I-8.*** MS data evaluation is more complex than MB or LCS data evaluation since MSs measure matrix effects in addition to sample preparation and analysis errors. The heterogeneity of soil, grab samples, and sequentially collected water samples further complicates the evaluation since matrix-specific bias assumes that the native concentrations in the duplicate analyses are constant. In addition concentrations of the target analytes in the sample can also far exceed the spike amounts added, making the resulting recoveries invalid. MSs that fail to meet the appropriate acceptance criteria would indicate that a potential matrix effect is present. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences between the native concentration of the unspiked sample and the spiked samples may not be significant, making the bias measures unrepresentative of the true method and matrix performance. ***For this reason, if the native concentration is two or more times the spiking level, corrective actions would be based on project DQOs.*** Regardless, steps should be taken to find the cause of failure and corrective actions be taken to remedy it. If possible, respike the sample as outlined in the following sections at a higher level (e.g., at two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. A review of the MSD result, if available, may confirm the matrix effect, if it is the same direction and same order of magnitude. If the native concentration is low, and the MS/MSD recoveries confirm matrix interference, reanalyze the MS/MSD sample/extract after employing cleanup procedures (organic analyses) or dilution techniques to minimize matrix interference. ***If the matrix effect cannot be resolved, discuss the impact on the data within the case narrative.***

I.11.4.3.1 Inorganic analyses. Corrective action for unacceptable MS recoveries for ICP and GFAA analyses shall include implementation of a PDS from the same sample that the MS was prepared. In that way, information is obtained to identify whether matrix interference is occurring during the digestion or analytical procedures. Refer to Section I.11.4.6 for guidance on the evaluation of MS in conjunction with the PDS.

I.11.4.3.2 Organic analyses. When multiple (>5) target analytes are reported, the acceptance criteria may allow for the sporadic marginal failure of a few target analytes included within the MS without requiring reanalysis. When only a subset of target analytes is included in the MS, allow only one sporadic marginal failure. Reference Section I.9.3 and Tables I-1 through I-8 for information on the number of sporadic failures allowed and the expanded acceptance criteria to be applied.

I.11.4.4 Matrix duplicate (MD) and matrix spike duplicate (MSD) samples. The MSD is evaluated using the same bias criteria as described for the MS. ***The MD or MSD is evaluated by comparing the***

precision for all target analytes to the windows as determined by project-specific DQOs, or as stated herein. These criteria should be applied only to concentrations of target analytes that are above the MQL of each analyte. MDs or MSDs that fail to meet the appropriate acceptance criteria would indicate that a potential matrix effect is present. Corrective actions shall be performed as described for the MS.

I.11.4.5 Surrogates. ***A surrogate is evaluated by comparing its recovery in each sample to the windows as determined by project-specific DQOs, or as stated within Tables I-3 through I-8.*** Surrogate spikes in matrix-specific samples that fail to meet the appropriate acceptance criteria would indicate that a potential matrix effect is present. If significant nontarget interference occurs, corrective action shall include implementing additional cleanup procedures and reanalyses. ***If this does not reduce the interference, discuss the impact on the data within the case narrative. Recommendations to the client may include method modifications, such as repreparation and reanalysis with smaller sample aliquots to reduce the effects of the matrix.*** The consequences to detection limits must also be considered in this instance. Surrogate failures in MBs or LCSs are indicative of a general method failure and should be thoroughly investigated as noted in Sections I.11.4.1 and I.11.4.2, respectively.

I.11.4.6 Post-digestion spike samples. Default recovery control limits for the PDS are noted in Tables I-1 and I-2. Similar to the MS, if historic data or information on native sample concentrations is available, the MS or PDS should be spiked at a concentration at least twice the native sample concentration for the following evaluation to be considered valid. Professional judgment should be used to determine the corrective action necessary when the MS recovery for an analyte fails but the PDS recovery passes. ***For instance, when the MS recovery fails because it falls below the lower control limit but the PDS recovery passes, confirmatory redigestion and reanalysis may not be required if allowed by project DQOs.*** When both the MS and PDS indicate matrix interference is present, the laboratory must attempt to correct for the interference by the use of method of standard additions, an internal standard technique for ICP (e.g., with yttrium), a different matrix modifier for GFAA, or different digestion or analytical procedures to achieve a representative result, before qualifying the sample for matrix interference. This does not apply to sporadic failures but rather to target analytes exhibiting out-of-control recoveries on consecutive batches. Also, verify overall batch control for the analysis by evaluation of the LCS.

I.11.5 Calculation errors. Reports shall be reissued if calculation or reporting errors are noted with any given data package. The case narrative shall clearly state the reason(s) for reissuance of the report.

I.11.6 Onsite audits. A corrective actions report shall be required that addresses any deficiencies noted during audits conducted. ***If corrective actions are needed for major deficiencies that would affect data quality, the laboratory should notify USACE of other projects that may be affected.***

I.12 Target Analyte Identification, Quantitation, and Confirmation

I.12.1 Target analyte identification. Employ procedures presented within the individual determinative methods for determining presence and identification of target analytes within samples. ***For GC/MS analyses and any samples containing extraneous peaks not associated with the calibration standards, a scan against a mass spectral library (typically ~75,000 compounds) may be performed for the purposes of tentative identification if warranted by project DQOs.*** Based upon the degree of match, evidence of similar pattern, and analyst professional judgment, compounds may be reported as Tentatively Identified Compounds (TICs) and the analytical values estimated. ***The necessity to perform this will depend on project-specific requirements. Recommend the use of TIC searches only in the early stages of site characterization on samples speculated as contaminated. Significant detections identified through TIC searches should require the inclusion of these compounds as project-specific target analytes. Future***

analyses shall require that calibration standards include these target analytes for more accurate quantitative determination of their result.

I.12.2 Target analyte quantitation. All samples shall be quantitated using the initial calibration curve, following procedures outlined within the determinative methods. Sample results that exceed the range of the initial calibration high standard must be diluted and reanalyzed, and sample analyte values reported below the MQL must be flagged as estimated quantities (i.e., J-flag). All dilutions must be applied to the sample results and reported accordingly. Solid samples are to be determined on a dry-weight basis. Sample target analyte values should be reported to three significant figures.

I.12.2.1 Inorganic analyses. Quantitative results are calculated using the mean value from the set of duplicate injections for Method 7010 or the mean value from multiple exposures for Method 6010. Also recommend the laboratory review the RPDs for duplicate injections/multiple exposures of samples exhibiting quantifiable concentrations. If the %RPD/%RSD is consistently >20 percent and highly variable for concentrations greater than the low-level calibration standard, corrective action should be taken. When matrix interference is suspected/confirmed, the use of method of standard additions must be used to calculate the sample result. The laboratory shall at a minimum use a series of three standard additions containing 50, 100, and 150 percent of the expected concentration. As outlined within the method, plot the absorbance of each solution at the concentration of the known standards. The concentration of the sample is then obtained from extrapolating the resulting line back to zero absorbance.

I.12.2.2 Organic analyses. The laboratory should make a reasonable attempt to correct for any matrix interference encountered. Dilutions should not be routinely used in preference to cleanup methods to address matrix interference. When matrix interference is present, samples should be processed using at least one cleanup method as outlined by the determinative method. Refer to Section I.6.8.2.2 for information on recommended cleanup methods. ***If the cleanup and reanalysis do not reduce the matrix interference, discuss the impact on the data within the case narrative.***

I.12.2.2.1 Method 8081. In general, multiple-component analytes are quantitated (via external calibrations) by comparing the areas (or heights) for the characteristic peaks to the areas (or heights) for the corresponding calibration peaks of the same retention time and shape. Quantitation may be performed using a number (i.e., three to five) of major peaks or the total peak area of the appropriate pattern as described in the method. For chlordane, quantitate the peaks of alpha-chlordane, gamma-chlordane, and heptachlor separately against the initial 3-point calibration curves and report the individual results. When the GC pattern of the residue resembles that of technical chlordane, quantitate for this. Since commercial BHC (which consists of a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes) may exhibit a wide variance in the percentage of the individual isomers present, quantitate and report the alpha-, beta-, gamma-, and delta-BHC isomers separately. For DDT, the 4,4'-isomers of DDT, DDE, and DDD are the predominant pesticides in the environment and are the isomers normally regulated by USEPA. Therefore, quantitate separately and report the pure 4,4'-isomers of DDT.

I.12.2.2.2 Method 8330. Due to the lack of resolution between 2,4-DNT and 2,6-DNT, and between 2-Am-DNT and 4-Am-DNT, quantitation of these compounds may be expressed as isomeric pairs.

I.12.3 Target analyte confirmation. Chromatography is a technique that relies upon the comparison of retention times between standards and unknown peaks for qualitative identification. Unless mass spectrometry is used as the detector, tentative identification is based solely on the retention time of an unknown peak falling within the prescribed retention time window of a known standard. ***In the absence of project-specific criteria, to minimize the possibility of incorrect identification (or false positives), confirmation***

shall be required for all chromatographic methods involving the analysis of single-component target analytes. Confirmation may be required for multicomponent analytes even though identification is achieved primarily through pattern recognition (i.e., PCBs, gasoline, etc.). For instance, PCB analysis requires second column confirmation when the Aroclor identification is in doubt, when a mixture of Aroclors are present, or when the pattern is weathered. It is recommended that confirmation techniques involve the use of another analytical technique (i.e., GC/MS), or a second dissimilar column. ***When project DQOs allow, a different type of detector may also be used.*** When the second dissimilar column is used, it shall be calibrated in the same manner as the primary column. After the target analyte has been identified, compare the primary and confirmatory results for agreement according to a method-prescribed criterion. Analytical results would normally be reported from the primary column unless interferences were noted. If quantitative results are reported from the confirmation column, the documentation from the analysis of all appropriate QC samples on the confirmation column shall also be required within the data package.

I.13 Data Reduction, Review, and Reporting

I.13.1 Data reduction. Data reduction procedures, whether performed by the instrument or manually, shall follow methodologies outlined within the laboratory SOP or analytical method. Project-specific variations of the general procedures, statistical approach, or formulas may be identified, depending on project-specific requirements. Automated procedures shall be verified as required by EPA's guidance on GALP (EPA 2185): all software shall be tested with a sample set of data to verify its correct operation via accurate capture, processing, manipulation, transfer, recording, and reporting of data.

I.13.2 Data review. All analytical data generated by the laboratory shall be extensively reviewed prior to report release to assure the validity of the reported data. This internal data evaluation process shall cover the areas of data generation, reduction, and a minimum three levels of documented review. For each level, the review process shall be documented using an appropriate checklist that is signed and dated by the reviewer. The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the data evaluation is essential in ensuring that data of known quality are generated consistently. All data generated and reduced shall follow well-documented in-house protocols.

I.13.2.1 Level 1 analyst review. Each analyst reviews the quality of his/her work based on an established set of guidelines. The review criteria as established in each method, in this guidance, or within the laboratory shall be used. This review shall, at a minimum, ensure the following:

- Sample preparation information is correct and complete.
- Analysis information is correct and complete.
- The appropriate SOPs have been followed.
- Analytical results are correct and complete.
- Raw data, including all manual integrations, have been correctly interpreted.
- QC samples are within established control limits.

- Special sample preparation and analytical requirements have been met.
- Data transfers were verified.
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, anomaly forms are complete, holding times are documented, etc.). Level 1 analyst review shall be documented by using a checklist and by the signature of the reviewer and date.

I.13.2.2 Level 2 peer review. Level 2 reviews shall be performed by a supervisor, another analyst, or data review specialist who has documentation that supports demonstration of performance for all areas for which he/she provides review. The function of this review is to provide an independent, complete peer review of the analytical batch data package. This review shall also be conducted according to an established set of guidelines and is structured to ensure the following:

- All appropriate laboratory SOPs have been referenced.
- Calibration data are scientifically sound, appropriate to the method, and completely documented.
- QC samples are within established guidelines.
- Qualitative identification of sample components is correct.
- Quantitative results, including calculations and any associated flags, are correct.
- Raw data, including manual integrations, have been correctly interpreted.
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented, nonconformance forms are complete, holding times are documented, etc.).
- The data are ready for incorporation into the final report.

Level 2 reviews shall be structured so that all calibration data and QC sample results are reviewed and all of the analytical results are checked back to the raw data or bench sheets. If no problems are found with the data package, the review is complete. If any problems are found with the data package, then all sample results shall be returned to the analyst and rechecked. All errors and corrections noted shall be documented. Level 2 peer reviews shall also be documented on a checklist with the signature of the reviewer and date.

I.13.2.3 Level 3 administrative review. Level 3 reviews are performed by the program administrator or designee at the laboratory. This review shall provide a total overview of the data package, including sample receipt, to ensure its consistency and compliance with project-specific requirements. All errors noted shall be corrected and documented. Based on the errors noted, samples may need to be reprepared and reanalyzed. Level 3 administrative reviews shall also be documented on a checklist with the signature of the reviewer and date.

I.13.2.4 QA review. QA review is performed by the QA Officer or QA Branch. This review is not part of the normal production data review process. The QA Officer would typically review at least 10 percent of the data produced by the laboratory using the procedures as outlined in the Level 3 data reviews. Additional technical details should be reviewed in this QA review, similar to Levels 1 and 2, along

with a total package review, i.e., correlation of results from differing but related chemical parameters. The data packages reviewed would be randomly selected by the QA Officer. Nonconformance reports would be required for any errors noted.

I.13.3 Data qualifiers. Data qualifiers shall be added by the laboratory during the data generation/ review process. These qualifiers will be applied when measurement quality objectives defined in Section I.11 are not met and corrective action is not successful or when corrective action is not performed. All flags used by the laboratory shall be defined completely within the chemical data reportable packages. The following example data qualifiers are suggested for use:

- U = Nondetect when analyte concentration is below MRL.
- J = Estimated concentration when analyte concentration falls below the MQL (i.e., lowest calibration standard).
- B = Blank contamination when any associated blanks are above one-half the MRL.
- Q = Data requires usability review due to the exceedance of method-specific holding times, calibration, or batch QC data associated with the samples does not meet stated measurement quality objectives.

These flags should also identify any suspected bias in the data, either low or high, and whether the estimation is related to the suspected identification (qualitative) or whether the value reported is an approximation (quantitative). ***The project manager or appropriate technical personnel shall be notified as soon as possible to discuss possible corrective actions should data be qualified. Additional data flagging may be performed by the USACE designee based upon overall project-specific requirements, through the use of external data review or validation.***

I.13.4 Data reporting requirements. The chemistry data package should contain enough information to demonstrate that the project data quality objectives have been fulfilled. In general, one should be able to determine the precision, bias, representativeness, comparability, and sensitivity of the data from information contained in the data package. This description applies to both primary and referee laboratory packages. The amount of information required to demonstrate attainment of DQOs depends upon the acceptable level of uncertainty for the intended data use. In general, the type of data package required will fall into one of four general categories: Screening, Definitive, Performance-Based, and Comprehensive. All reported data packages must be retained by the laboratory for a minimum of five (5) years, or as dictated by project requirements (if longer than five years). In the event of laboratory closure, all applicable documents must be transferred to the USACE client.

I.13.4.1 Screening data package. Screening data are generated by methods of analysis that tend to be relatively rapid, are performed in the field (as opposed to an offsite laboratory), and may have less rigorous sample preparation. Screening data provide analyte identification but may tend to report false positives. Their ability to quantitate analytes is in general less precise and less accurate than "definitive" type methods (see next section). Screening data must be confirmed by sending at least 10 percent of the samples for definitive analysis. The screening data package will depend on the screening method used. A typical screening data package will include the following:

- Sample identification number
- Preparation method

- Determinative method
- Detection limits
- Identity and quantity of analyte(s) present
- Date and time of sample collection
- Date of sample analysis
- Field equipment calibration

More sophisticated field screening methods will involve QC samples such as duplicate samples, calibration standards, spiked samples, or blank samples. Results for these associated QC samples should also be included in the screening data package.

I.13.4.2 Definitive data package. The definitive data package format allows for the review of the data by an independent organization. However, this data package does not allow for complete independent reconstruction of the analytical data. Definitive data are produced using rigorous analytical methods, such as USEPA standard reference methods (e.g., SW-846, CLP). Analyte presence and quantitation are confirmed through extensive quality control procedures at the laboratory, which may be onsite or offsite. As discussed in more detail in the following sections, the definitive data package should include a cover sheet, table of contents, case narrative, the analytical results, laboratory reporting limits, sample management records, and internal laboratory QA/QC information. The laboratory data package should be organized such that the analytical results are reported on a per-batch basis unless otherwise specified.

I.13.4.2.1 Cover sheet. The cover sheet should specify the following information:

- Title of report (i.e., Test Report, Test Certificate).
- Name and location of laboratory (to include a point of contact, phone and facsimile numbers).
- Name and location of any subcontractor laboratories, and appropriate test method performed.
- Contract number.
- Client name and address.
- Project name and site location.
- Statement of data authenticity and official signature and title of person authorizing report release.
- Amendments to previously released reports shall clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report.

I.13.4.2.2 Table of contents. Laboratory data packages should be organized in a format that allows for easy identification and retrieval of information. An index or table of contents should be included for this purpose.

I.13.4.2.3 Case narrative. A case narrative should be included in each report. The case narrative should contain a table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed and by which laboratories. Samples that were received but not analyzed should also be identified. Extractions or analyses that are performed out of holding times should be appropriately noted. The case narrative should define all data qualifiers or flags used. Deviations of any calibration standards or QC sample results from appropriate acceptance limits should be noted and associated corrective actions taken by the laboratory should be discussed. Any other factors that could affect the sample results (e.g., air bubbles in VOC sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature and sample pH excursions, container type or volume, etc.) should be noted.

I.13.4.2.4 Analytical results. The results for each sample should contain the following information at a minimum. (Information need not be repeated if noted elsewhere in the data package).

- Laboratory name and location (city and state).
- Project name and unique ID number.
- Field sample ID number as written on custody form.
- Laboratory sample ID number.
- Matrix (soil, water, oil, etc.).
- Sample description.
- Sample preservation or condition at receipt.
- Date sample collected.
- Date sample received.
- Date sample extracted or prepared.
- Date sample analyzed.
- Analysis time when holding time limit <48 hours.
- Method (and SOP) numbers for all preparation, cleanup, and analysis procedures employed.
- Preparation, analysis, and other batch numbers.
- Analyte or parameter.
- Method reporting limits adjusted for sample-specific factors (e.g., aliquot size, dilution/ concentration factors, moisture content).
- Method quantitation limits (low-level standard concentration).
- Method detection limits.
- Analytical results with correct number of significant figures.
- All confirmation data.

- Any data qualifiers assigned.
- Concentration units.
- Dilution factors. All reported data shall reflect any dilutions or concentrations. The dilution factor, if applicable, should be noted on the analytical report. If neat and/or diluted results are available, data from all runs should be recorded and reported.
- Percent moisture or percent solids (all soils, sediments, sludges, etc. are to be reported on a dry weight basis).
- Chromatograms, as needed.
- Sample aliquot analyzed.
- Final extract volume.

I.13.4.2.5 Laboratory reporting limits. The laboratory may use a reporting limit expressed in terms of detection limit, quantitation limit, regulatory action level, or project-specific threshold limits. However, the laboratory's use of these terms must be well defined. In addition, the "<" reporting convention must be used in accordance with the requirements established in Section I.3.3.7.3.

I.13.4.2.6 Sample management records. These types of records include the documentation accompanying the samples (i.e., original chain-of-custody record, shipping documents, laboratory notification sheets), records generated by the laboratory that detail the condition of the samples upon receipt at the laboratory (i.e., sample cooler receipt forms, any telephone conversation records, etc.), and any records generated to document sample custody, transfer, analysis, and disposal.

I.13.4.2.7 QA/QC information. The minimum data package must include the calibration, calibration verification, and internal laboratory QA/QC data with their respective acceptance criteria. The data package should also include the laboratory's method quantitation and reporting limits for project-specific parameters. The calibration data shall include a summary of the ICV, all calibration verification standards, and any performance standards analyzed in conjunction with the test method. All calibration deviations shall be discussed within the case narrative. The data package should correlate the method QC data with the corresponding environmental samples on a per-preparation batch basis with batch numbers clearly shown. Method QC data must include all spike target concentration levels; the measured spike concentration and calculated recoveries; all measures of precision, including relative percent difference; and all control limits for bias and precision. This would include laboratory performance information such as results for MBs, recoveries for LCSs, and recoveries for QC sample surrogates; and matrix-specific information such as MD RPDs, MS and MSD recoveries, MS/MSD RPDs, field sample surrogate recoveries, SDs, and PDS, etc. At a minimum, internal QC samples should be analyzed and reported at rates specified in the specific methods, within USACE guidance, or as specified in the contract, whichever is greater. Any deviations from the measurement quality objectives should be noted. Also include any data review, nonconformance, or corrective action forms within the data package.

I.13.4.3 Performance-based data package. The requirements for the ***performance-based data package are the same as those defined within the definitive data package with the addition of the following items: all appropriate project action level(s) and DQOs and appropriate preparatory and analysis logs.***

I.13.4.4 Comprehensive data package. A comprehensive data package contains sufficient information to completely reconstruct the chemical analyses that were performed. Hence, comprehensive data packages include all batch QC results, instrument QC results (e.g., initial calibration verification, continuing calibration verification, and instrument performance checks), MDL studies, and raw data (e.g., run logs, sample preparation logs, standard preparation logs, and printed instrumental output such as chromatograms). Typically, comprehensive data packages are required if third-party data validation is to be performed. The data validation guidelines for performance-based methods established in other USACE guidance on data review and data validation, USEPA national functional guidelines, USEPA regional functional guidelines, and project-specific guidelines for validation may all have distinct reporting formats. The appropriate validation guidelines should be consulted to determine what type of data package is required.

I.13.4.5 Chemistry data package deliverable time schedule. A schedule for data delivery should be established so that data packages are provided as needed for chemical QA assessment. This includes identifying the anticipated number or frequency of these data packages in light of project objectives, i.e., the amount of data produced or project duration.

I.13.4.6 Electronic data deliverables. Electronic data deliverables (EDDs) may be specified either in addition to or in lieu of hard copy requirements. EDDs shall contain the same information as described for the hard copy deliverables. The complete set of rules for representing these data in a form suitable for transmission is called an EDD format. EDDs should use a common syntax for terms used to describe diverse laboratory activities and report analytical data. EDDs should also provide sufficient input parameters to allow users to link analytical data to underlying laboratory activities, full traceability for data, and a means for reporting complex analytical relationships. Examples of EDDs include DEEMS, IRPMIS, COELT, etc. DEEMS (Department of Energy Environmental Management Electronic Data Deliverable Master Specification) is based on a sophisticated model of analytical activities and uses a flexible way of representing the linkages between these activities. DEEMS is the recommended EDD for USACE projects when electronic data are specified. Information on the availability of this DEEMS implementation guide may be obtained from the Chemical Data Quality Management Branch, Hazardous, Toxic, and Radioactive Waste Center of Expertise, U.S. Army Corps of Engineers.

Table I-1
Summary of Measurement quality objectives for Method 6010 Inductively Coupled Plasma (ICP) Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (I.9.2.1.1)	Option 1- 1 std and blank, and a low-level check standard at MQL Option 2- 3 stds and blank	Daily	Option 1- Low-level check standard ± 20% Option 2- r • 0.995
Instrumental Precision (I.9.2.1.1)	%RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	%RSD < 5%
Initial Calibration Verification (ICV) (I.9.3)	Midlevel (2nd source) verification	After initial calibration	%Recovery ± 10%
Initial Calibration Blank (ICB) (I.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL
Interelement Check Standards (ICS) (I.8.1)	ICS-A - interferents only ICS-B - interferents and target analytes	Beginning of analytical sequence	%Recovery ± 20% for target analytes
Continuing Calibration Blank (CCB) (I.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL
Continuing Calibration Verification (CCV) (I.9.5 / I.9.5.1)	Midlevel verification	Every 10 samples and at end of analytical sequence	%Recovery ± 10%
Method Blank (MB) (I.10.2.1 / I.11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < one-half MRL
Laboratory Control Sample (LCS) (I.10.2.2 / I.11.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	%Rec = 80% - 120% Sporadic marginal failures[1]: %Rec = 60% - 140%
Matrix Spike (MS) (I.10.2.3 / I.11.4.3 / I.11.4.3.1)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	%Rec = 75% - 125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (I.10.2.4 / I.11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD • 25%
Post Digestion Spike (PDS) (I.10.3.1 / I.11.4.6)	Sample digestate spiked with all/subset of target analytes	1 per sample batch on MS sample	%Rec = 75% - 125%
Serial Dilution (SD) (I.10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Additions (MSA) (I.12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r • 0.995

[1] The number of Sporadic Marginal Failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed.

Table I-2
Summary of Measurement quality objectives for Method 7010/7470/7471 Series GFAA/CVAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (I.9.2.1.2)	3 stds and blank(GFAA) 5 stds and blank(CVAA)	Daily	r • 0.995
Instrumental Precision (I.9.2.1.2)	RPD of 2 injections	All standards, and ICV/CCV	RPD ± 10%
Initial Calibration Verification (ICV) (I.9.3)	Midlevel (2nd source) verification	After initial calibration	%Rec ± 10%
Initial Calibration Blank (ICB) (I.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL
Continuing Calibration Blank (CCB) (I.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL
Continuing Calibration Verification (CCV) (I.9.5 / I.9.5.1)	Midlevel verification	Every 10 samples and at end of analytical sequence	%Rec ± 20%
Method Blank (MB) (I.10.2.1 / I.11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < one-half MRL
Laboratory Control Sample (LCS) (I.10.2.2 / I.11.4.2)	Interference-free matrix containing target analytes	1 per sample batch	%Rec = 80% - 120%
Matrix Spike (MS) (I.10.2.3 / I.11.4.3 / I.11.4.3.1)	Sample matrix spiked with target analytes prior to digestion	1 per sample batch	%Rec = 80% - 120%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (I.10.2.4 / I.11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD • 20%
Post Digestion Spike (PDS) (I.10.3.1 / I.11.4.6)	Sample digestate spiked with target analytes	Every sample	%Rec = 85% - 115%
Serial Dilution (SD) (I.10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Additions (MSA) (I.12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r • 0.995

Note: GFAA = Graphite furnace - atomic absorption spectroscopy.
CVAA = Cold vapor - atomic absorption.

Table I-3
Summary of Measurement quality objectives for Method 8021 VOCs

QC Element	Target Analyte/Surrogate	Poor Purgers/Gases/Sporadic Marginal Failures[1]
Initial Calibration (I.9.2.2.1)	Primary Evaluation: r • 0.995, %RSD • 20%, r^2 • 0.990	No allowance
	Alternative Evaluation: Mean %RSD for all target analytes • 20%	Alternative Evaluation: Maximum allowable %RSD for each target analyte • 40%
ICV (I.9.3)	%Rec = 85% - 115%	No allowance
CCV (I.9.5 / I.9.5.2 / I.9.5.2.1)	Primary Evaluation: %Drift • 15%, %D • 15%	Primary Evaluation: %Drift • 20%, %D • 20%
	Alternative Evaluation: Mean %Drift/%D for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %Drift/%D for each individual target analytes • 30%
MB (I.10.2.1 / I.11.4.1)	Target Analytes: Analytes < one-half MRL	Common Lab Contaminants: Analytes < MRL
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	%Rec = 70% - 130%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
MSD/MD (I.10.2.4 / I.11.4.4)	Water: RPD • 30% Solids: No RPD Limits	Water: RPD • 40% Solids: No RPD Limits
Surrogates (I.10.2.5 / I.11.4.5)	Interference-Free Matrix: Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125% Project Sample Matrix: %Rec = 70% - 130%	Not applicable
Target Analyte Confirmation (I.12.3)	RPD • 40%	RPD • 40%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if the 8020 Target Analyte List (10 compounds) is reported, 1 SMF is allowed. If the 8010 Target Analyte List (32 compounds) is reported, 3 SMFs are allowed. If the full 8021 Target Analyte List (60 compounds) is reported, 4 SMFs are allowed. If the MS includes only a subset of compounds, allow only 1 SMF for that QC element..

Table I-4
Summary of Measurement quality objectives for Method 8081 Pesticides

QC Element	Target Analyte/Surrogate	Sporadic Marginal Failure [1]
DDT/Endrin %Breakdown (I.8.2)	DDT & Endrin %Breakdown • 15% each	Not Applicable
Initial Calibration (I.9.2.2.4)	Primary Evaluation: r • 0.995, %RSD • 20%, r^2 • 0.990	No allowance
	Alternative Evaluation: Mean %RSD for all target analytes • 20%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %RSD for each individual target analyte • 40%
ICV (I.9.3 / I.9.3.1)	%Rec = 85% - 115%	No allowance
CCV (I.9.5 / I.9.5.2 / I.9.5.2.2)	Primary Evaluation: %Drift • 15%, %D • 15%	No allowance
	Alternative Evaluation: Mean %Drift/%D for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %Drift/%D for each individual target analyte • 30%
MB (I.10.2.1 / I.11.4.1)	Analytes < one-half MRL	Not applicable
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130%	Sporadic Marginal Failures[1]: %Rec = 30% - 150%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	%Rec = 40% - 140%	Sporadic Marginal Failures[1]: %Rec = 30% - 150%
MSD/MD (I.10.2.4 / I.11.4.4)	RPD • 50%	RPD • 60%
Surrogates (I.10.2.5 / I.11.4.5)	Interference-Free Matrix: Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130% Project Sample Matrix: %Rec = 40% - 140%	Not applicable
Target Analyte Confirmation (I.12.3)	RPD • 40%	RPD • 40%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if the full list of 21 compounds is reported from the GC/electron capture detector analysis, then 2 SMFs are allowed to the expanded criteria presented. If the MS includes only a subset of compounds, allow only 1 SMF for that QC element.

Table I-5
Summary of Measurement quality objectives for Method 8082
PCBs

QC Element	Target Analyte/Surrogate
Initial Calibration (I.9.2.2.5)	r • 0.995, RSD • 20%, r^2 • 0.990
ICV (I.9.3 / I.9.3.2)	%Rec = 85% - 115%
CCV (I.9.5 / I.9.5.2)	%Drift • 15%, %D • 15%
MB (I.10.2.1 / I.11.4.1)	Analytes < one-half MRL
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130%
MS (I.10.2.3 / I.11.4.3)	%Rec = 40% - 140%
MSD/MD (I.10.2.4 / I.11.4.4)	RPD = 50%
Surrogates (I.10.2.5 / I.11.4.5)	Interference-Free Matrix: Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130% Project Sample Matrix: %Rec = 40% - 140%
Target Analyte Confirmation (I.12.3)	RPD • 40%

Table I-6
Summary of Measurement quality objectives for Method 8260 VOCs

QC Element	Target Analyte/Surrogate	Poor Purgers/Gases/Sporadic Marginal Failures[1]
Initial Calibration (I.9.2.2.4)	Instrument Evaluation: System performance check compounds (SPCCs): minimum response factor (RF) values per method requirements Calibration check compounds (CCCs): verify %RSD • 30%	No allowance
	Primary Evaluation: r • 0.995, %RSD • 15%, r^2 • 0.990	No allowance
	Alternative Evaluation: Mean %RSD for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %RSD for each individual target analyte • 30%
ICV (I.9.3)	%Rec = 80% - 120%	No allowance
CCV (I.9.5 / I.9.5.2 / I.9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements	No allowance
	Primary Evaluation (CCCs): %Drift • 20%, %D • 20%	No allowance
MB (I.10.2.1 / I.11.4.1)	Target Analytes: Analytes < one-half MRL	Common Lab Contaminants: Analytes < MRL
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	%Rec = 70% - 130%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
MSD/MD (I.10.2.4 / I.11.4.4)	Water: RPD • 30% Solids: No RPD Limits	Water: RPD • 40% Solids: No RPD Limits
Surrogates (I.10.2.5 / I.11.4.5)	%Interference-Free Matrix: Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125% Project Sample Matrix: %Rec = 70% - 130%	Not applicable

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if the full list of 68 compounds is reported from the GC/MS analysis, then 5 SMFs are allowed to the expanded criteria presented for the LCS. If the MS includes only a subset of compounds, allow only 1 SMF for this QC element.

Table I-7
Summary of Measurement quality objectives for Method 8270 Semivolatiles

QC Element	Target Analyte/Surrogate	Poor Performers/Sporadic Marginal Failures[1]
Initial Calibration (I.9.2.2.7)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD • 30%	No allowance
	Primary Evaluation (all target analytes) : r • 0.995, %RSD • 15%, r^2 • 0.990	No allowance
	Alternative Evaluation: Mean %RSD for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %RSD for each individual target analyte • 40%
ICV (I.9.3)	%Rec = 70% - 130%	No allowance
CCV (I.9.5 / I.9.5.2 / I.9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements	No allowance
	Primary Evaluation (CCCs): %Drift • 20%, %D • 20%	No allowance
MB (I.10.2.1 / I.11.4.1)	Target Analytes: Analytes < one-half MRL	Common Lab Contaminants: Analytes < MRL
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 60% - 120% (~15 analytes) = 45% - 135% (~30 analytes) = 20% - 150% (~15 analytes) Solids: %Rec = 60% - 120% (~20 analytes) = 45% - 135% (~25 analytes) = 30% - 150% (~15 analytes)	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 25% - 150%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	Water: %Rec = 45% - 135% Solids: %Rec = 45% - 135%	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%
MSD/MD (I.10.2.4 / I.11.4.4)	Water: RPD • 50% Solids: RPD • 60%	Sporadic Marginal Failures[1]: Water: RPD • 60% Solids: RPD • 60%
Surrogates (I.10.2.5 / I.11.4.5)	%Interference-Free Matrix[2]: Water: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Solids: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Project Sample Matrix:[2] Water: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds Solids: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if the full list of target compounds as presented in Table 13 are reported, then 5 SMFs are allowed to the expanded criteria presented for the LCS. If the MS includes only a subset of compounds and for Surrogates, allow up to 1 SMF.
[2] B = base, N = neutral, and A = acid compounds (cmpds).

Table I-8
Summary of measurement quality objectives for Method 8330 Explosives

Initial Calibration (I.9.2.2.8)	Primary Evaluation: r • 0.995, RSD • 20%, r^2 • 0.990 Alternative Evaluation: Mean %RSD for all target analytes • 20%, with maximum allowable restriction noted at right for individual analytes.	No allowance Alternative Evaluation: Maximum allowable %RSD for each individual target analyte • 40%
ICV (I.9.3)	%Rec = 85% - 115%	No allowance
CCV (I.9.5 / I.9.5.2)	Primary Evaluation: %Drift • 15%, %D • 15% Alternative Evaluation: Mean %Drift/%D for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	No allowance Alternative Evaluation: Maximum allowable %Drift/%D for each individual target analyte • 30%
MB (I.10.2.1 / I.11.4.1)	Target Analytes: Analytes < one-half MRL	Not applicable
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 60% - 120%[2] Solids:%Rec = 60% - 120%[2]	Sporadic Marginal Failures[1]: %Rec = 40% - 150%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	%Rec = 50% - 140%[2]	Sporadic Marginal Failures[1]: %Rec = 40% - 150%
MSD/MD (I.10.2.4 / I.11.4.4)	RPD • 50%	RPD • 60%
Surrogates (I.10.2.5 / I.11.4.5)	Interference-Free Matrix: Water: %Rec = 60% - 140% Solids: %Rec = 50% - 150% Project Sample Matrix: %Rec = 50% - 150%	Not applicable
Target Analyte Confirmation (I.12.3)	RPD • 40%	RPD • 40%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 explosives are reported from the high-performance liquid chromatography analysis, 1 SMF is allowed to the expanded criteria presented for the LCS. If greater than 15 explosives are reported, 2 SMFs are allowed for the LCS. If the MS includes only a subset of compounds, allow only 1 SMF for this QC element.
[2] Due to the tendency for Tetryl to decompose, an expanded criteria may be applied at 45% - 140% for both water and soil matrices.

Table I-9
Target Analyte List for Method 8021 VOCs

Target Analyte	Chemical Abstract Service (CAS) Registry No.
Benzene [1, 2]	71-43-2
Bromobenzene [3]	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane [3]	75-27-4
Bromoform [3]	75-25-2
Bromomethane [3, 4]	74-83-9
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon tetrachloride [3]	56-23-5
Chlorobenzene [1, 3]	108-90-7
Chloroethane [3, 4]	75-00-3
Chloroform [3]	67-66-3
Chloromethane [3, 4]	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane [3]	124-48-1
1,2-Dibromo-3-chloropropane [5]	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane [3]	74-95-3
1,2-Dichlorobenzene [1, 3]	95-50-1
1,3-Dichlorobenzene [1, 3]	541-73-1
1,4-Dichlorobenzene [1, 3]	106-46-7
Dichlorodifluoromethane [3, 4]	75-71-8
1,1-Dichloroethane [3]	75-34-3
1,2-Dichloroethane [3]	107-06-2
1,1-Dichloroethene [3]	75-35-4
cis-1,2-Dichloroethene	156-59-2

(Continued)

[1] AVO target analytes.
[2] BTEX target analyte list.
[3] HVO target analytes.
[4] Gaseous target analyte.
[5] Exhibits poor purging efficiency or instrumental response.

Table I-9 (Concluded)

Target Analyte	CAS Registry No.
trans-1,2-Dichloroethene[3]	156-60-5
1,2-Dichloropropane[3]	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene[3]	10061-01-5
trans-1,3-Dichloropropene[3]	10061-02-6
Ethyl Benzene[1,2]	100-41-4
Hexachlorobutadiene	87-68-3
Isopropylbenzene (Cumene)	98-82-8
p-Isoproplytoluene (p-Cumene)	99-87-6
Methylene chloride[3]	75-09-2
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane[3]	630-20-6
1,1,2,2-Tetrachloroethane[3]	79-34-5
Tetrachloroethene[3]	127-18-4
Toluene[1,2]	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane[3]	71-55-6
1,1,2-Trichloroethane[3]	79-00-5
Trichloroethene (trichloroethylene)[3]	79-01-6
Trichlorofluoromethane[3,4]	75-69-4
1,2,3-Trichloropropane[3]	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride[3,4]	75-01-4
o-Xylene[1,2]	95-47-6
m-Xylene[1,2]	108-38-3
p-Xylene[1,2]	106-42-3

[1] AVO target analytes.
[2] BTEX target analyte list.
[3] HVO target analytes.
[4] Gaseous target analyte.

Table I-10
Target Analyte List For Method 8081 Pesticides

Target Analyte	CAS Registry No.
Aldrin	309-00-2
alpha-BHC	319-84-6
beta-BHC	319-85-7
gamma-BHC (Lindane)	58-89-9
delta-BHC	319-86-8
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2

Table I-11
Target Analyte List for Method 8082 PCBs

Target Analyte	CAS Registry No.
As Aroclors	
Aroclor-1016	12674-11-2
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5
Congeners	
2-Chlorobiphenyl	2051-60-7
2,3-Dichlorobiphenyl	16605-91-7
2,2',5-Trichlorobiphenyl	37680-65-2
2,4',5-Trichlorobiphenyl	16606-02-3
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6
2,2',3,4,4',5, 5'-Heptachlorobiphenyl	35065-29-3
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9

Table I-12
Target Analyte List for Method 8260 VOCs

Target Analyte	CAS Registry No.
Acetone [1]	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane [1]	74-83-9
2-Butanone (methyl ethyl ketone) [1]	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon disulfide [1]	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane [1]	75-00-3
Chloroform	67-66-3
Chloromethane [1]	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane [1]	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane [1]	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6

(Continued)

[1] Denotes poor purging efficiency or poor response.

Table I-12 (Concluded)

Target Analyte	CAS Registry No.
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethyl Benzene	100-41-4
Hexachlorobutadiene	87-68-3
2-Hexanone [1]	591-78-6
Iodomethane	74-88-4
Isoproplybenzene (Cumene)	98-82-8
p-Isoproplytoluene (p-Cumene)	99-87-6
Methylene chloride	75-09-2
4-Methyl-2-pentanone [1]	108-10-1
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene (trichloroethylene)	79-01-6
Trichlorofluoromethane [1]	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride [1,2]	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

[1] Denotes poor purging efficiency or poor response.
[2] Gaseous target analyte.

Table I-13
Target Analyte List For Method 8270

Target Analyte	CAS Registry No.
Base/Neutral Fraction	
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Acetophenone	98-86-2
Aniline [1]	62-53-3
Anthracene	120-12-7
Benzidine [1]	92-87-5
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Benzyl alcohol [1]	100-51-6
4-Bromophenyl phenyl ether	101-55-3
Butyl benzyl phthalate	85-68-7
4-Chloroaniline [1]	106-47-8
bis(2-Chloroethoxy)methane	111-91-1
bis(2-Chloroethyl) ether	111-44-4
bis(2-Chloroisopropyl) ether	108-60-1
2-Chloronaphthalene	91-58-7
4-Chlorophenyl phenyl ether	7005-72-3
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Di-n-butyl phthalate	84-74-2
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
3,3'-Dichlorobenzidine	91-94-1
Diethyl phthalate [1]	84-66-2
Dimethyl phthalate	131-11-3

[1] Denotes poor extraction efficiency, tendency to decompose, or poor chromatographic behavior.

(Sheet 1 of 3)

Table I-13 (Continued)

Target Analyte	CAS Registry No.
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Di-n-octyl phthalate	117-84-0
Diphenyl amine	122-39-4
1,2-Diphenylhydrazine	122-66-7
bis(2-Ethylhexyl) phthalate	117-81-7
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene [1]	77-47-4
Hexachloroethane	67-72-1
Hexachloropropene	1888-71-7
Indeno(1,2,3-cd)pyrene	193-39-5
Isophorone	78-59-1
2-Methylnaphthalene	91-57-6
Naphthalene	91-20-3
2-Naphthylamine	91-59-8
2-Nitroaniline [1]	88-74-4
3-Nitroaniline [1]	99-09-2
4-Nitroaniline [1]	100-01-6
Nitrobenzene	98-95-3
N-Nitroso-dimethylamine [1]	62-75-9
N-Nitrosodiphenylamine [1,2]	86-30-6
N-Nitroso-di-n-propylamine	621-64-7
N-Nitrosopyrrolidine	930-55-2
Phenanthrene	85-01-8
Pyrene	129-00-0
Pyridine	110-86-1
1,2,4,5-tetrachlorobenzene	95-94-3
1,2,4-Trichlorobenzene	120-82-1

[1] Denotes poor extraction efficiency, tendency to decompose, or poor chromatographic behavior.
[2] N-Nitrosodiphenylamine coelutes with and cannot be differentiated from diphenylamine.

Table I-13 (Concluded)
Acid Fraction Target Analyte List For Method 8270

Target Analyte	CAS Registry No.
Acid Fraction	
Benzoic Acid [1]	65-85-0
4-Chloro-3-methylphenol [1]	59-50-7
2-Chlorophenol	95-57-8
2,4-Dichlorophenol	120-83-2
2,6-Dichlorophenol	87-65-0
2,4-Dimethylphenol [1]	105-67-9
4,6-Dinitro-2-methylphenol [1]	534-52-1
2,4-Dinitrophenol [1]	51-28-5
2-Methylphenol [1] (o-cresol)	95-48-7
3-Methylphenol [1,3] (m-cresol) & 4-Methylphenol [1,3] (p-cresol)	108-39-4 & 106-44-5
2-Nitrophenol [1]	88-75-5
4-Nitrophenol [1]	100-02-7
Pentachlorophenol [1]	87-86-5
Phenol [1]	108-95-2
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

[1] Denotes poor extraction efficiency, tendency to decompose, or poor chromatographic behavior.
[3] 3-Methylphenol (m-cresol) coelutes with 4-Methylphenol (p-cresol). Therefore, both are reported as isomeric pairs.

(Sheet 3 of 3)

Table I-14
Target Analytes For Method 8330 Explosives

Target Analyte	CAS Registry No.
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
1,3-Dinitrobenzene (1,3-DNB)	99-65-0
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8
Nitrobenzene (NB)	98-95-3
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	1946-51-0
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	355-72-78-2
2,4-Dinitrotoluene (2,4-DNT)	121-14-2
2,6-Dinitrotoluene (2,6-DNT)	606-20-2
2-Nitrotoluene (2-NT)	88-72-2
3-Nitrotoluene (3-NT)	99-08-1
4-Nitrotoluene (4-NT)	99-99-0

Table I-15
Number of Allowable QC Failures

N	X
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

Note: N = number of reported method target analytes
X = sporadic marginal failures allowed

Appendix J
Sampling and Analysis Plan Review Checklist

Sampling and Analysis Plan (SAP)
Review Checklist

Project Name: _____

Project Location: _____

GENERAL

Title Page
- a. Is project title listed? Y__N__N/A__
- b. Are names of principal investigators listed? Y__N__N/A__
- c. Are approval/signature lines for responsible parties listed? Y__N__N/A__
- d. Are abbreviations and acronyms listed? Y__N__N/A__

Table of Contents
- a. Is list of essential elements present? Y__N__N/A__
- b. Is list of figures present? Y__N__N/A__
- c. Is list of tables present? Y__N__N/A__
- d. Is list of appendices present? Y__N__N/A__

FIELD SAMPLING PLAN

Project Description
(This information may be referenced to the Project Work Plan.)
- a. Is site location/description discussed? Y__N__N/A__
- b. Is site map present? Y__N__N/A__
- c. Is site history discussed? Y__N__N/A__
- d. Is description of soils, geology, and hydrogeology at site discussed? Y__N__N/A__
- e. Are previous investigations/reports described? Y__N__N/A__

Project Organization and Responsibilities
(This information may be referenced to the Project Work Plan.)
- a. Is responsible organization identified? Y__N__N/A__
- b. Are subcontractors identified? Y__N__N/A__
- c. Are lines of authority identified? Y__N__N/A__

Scope and Objectives of the Field Investigation
- a. Is the purpose of the investigation described? Y__N__N/A__
- b. Are the objectives of the investigation identified for each medium of concern? Y__N__N/A__
- c. Are background data summarized? Y__N__N/A__
- d. Are data gaps identified for each medium? Y__N__N/A__
- e. Are the specific uses of the data (regulatory, risk assessment, etc.) identified? Y__N__N/A__
- f. Is a chart with regulatory/risk-based decision criteria included to ensure appropriate methods and reporting limits are used? Y__N__N/A__

Field Investigation Rationale
- a. Is rationale for geophysical investigations identified? Y__N__N/A__
- b. Are summary figures/tables identifying sampling locations/ analytical analyses by medium included? Y__N__N/A__
- c. Groundwater investigation
  - 1. Is the rationale for monitoring well locations clear? Y__N__N/A__

2. Are upgradient wells or background well locations included? Y__N__N/A__
3. Will well locations define vertical and horizontal extent of contamination? Y__N__N/A__
4. Is the rationale for the well depth/screen depth discussed? Y__N__N/A__
5. Is the rationale for slug tests/pump tests discussed? Y__N__N/A__
6. Is the rationale for the sampling locations/sampling frequency and type of analyses and measurements discussed? Y__N__N/A__
7. Is the rationale and frequency for the QC samples discussed? Y__N__N/A__
8. Are QC samples required to be associated with critical samples? Y__N__N/A__

d. Subsurface Soil Investigations
1. Is the rationale for soil boring locations clear? Y__N__N/A__
2. Are background soil borings included? Y__N__N/A__
3. Will soil borings define vertical and horizontal extent of contamination? Y__N__N/A__
4. Is the rationale for geophysical testing discussed? Y__N__N/A__
5. Is the rationale for the sampling locations/sampling frequency and type of analyses discussed? Y__N__N/A__
6. Are soil samples for geotechnical analysis discussed? Y__N__N/A__
7. Are field screening techniques described? Y__N__N/A__
and criteria identified? Y__N__N/A__
8. Are the rationale and frequency for the QC samples discussed? Y__N__N/A__
9. Are QC samples required to be associated with critical samples? Y__N__N/A__

e. Surface Soil Investigation
1. Is the rationale for the soil sampling locations clear? Y__N__N/A__
2. Is a soil sampling grid defined? Y__N__N/A__
3. Will the soil sampling locations define the horizontal extent of contaminations? Y__N__N/A__
4. Are background soil samples included? Y__N__N/A__
5. Is the rationale for the sampling locations/sampling frequency and type of analyses discussed? Y__N__N/A__
6. Are field screening techniques described? Y__N__N/A__
criteria identified? Y__N__N/A__
7. Are the rationale and frequency for the QC samples discussed? Y__N__N/A__
8. Are QC samples required to be associated with critical samples? Y__N__N/A__

f. Sediment Investigation
1. Is the rationale for the sediment sampling locations clear? Y__N__N/A__
2. Are background sediment samples included? Y__N__N/A__
3. Are samples colocated with SW samples, if needed for risk assessment? Y__N__N/A__
4. Will the sediment samples define the extent of contamination? Y__N__N/A__
5. Is the rationale for the sampling frequency and type of analyses discussed? Y__N__N/A__
6. Are field screening techniques described? Y__N__N/A__
and criteria identified? Y__N__N/A__
7. Are the rationale and frequency for the QC samples discussed? Y__N__N/A__
8. Are QC samples required to be associated with critical

samples? Y__N__N/A__

g. Surface Water Investigation
1. Is the rationale for the surface water sampling locations clear? Y__N__N/A__
2. Are background samples included? Y__N__N/A__
3. Are samples colocated with sediment samples, if needed for risk assessment? Y__N__N/A__
4. Will the surface water samples define the extent of contamination? Y__N__N/A__
5. Is the rationale for the sampling locations/sampling frequency and type of analyses discussed? Y__N__N/A__
6. Are field screening techniques described? Y__N__N/A__
and criteria identified? Y__N__N/A__
7. Are the rationale and frequency for the QC samples discussed? Y__N__N/A__
8. Are QC samples required to be associated with critical samples? Y__N__N/A__

Specific Field Investigation Activities/Procedures
a. Is a summary table of requirements for sample containers, preservation methods, holding time, and sample quantities presented? Y__N__N/A__
b. Drilling/Well Installation
1. Is the drilling method specified? Y__N__N/A__
2. Will the auger/drill stem and rig be decontaminated between holes? Y__N__N/A__
3. Is the length of the well screen defined? Y__N__N/A__
4. Is well screen placement consistent with contaminant location? Y__N__N/A__
5. Are the materials used for the well screen and casing consistent with contaminant type? Y__N__N/A__
6. Is thickness of well casing adequate for depth of well installation? Y__N__N/A__
7. Is a typical well diagram provided? Y__N__N/A__
8. Is there a minimum of 5 cm (2 in.) of annular space around the screen? Y__N__N/A__
9. Is screen slot size appropriate for the size? Y__N__N/A__
10. Does filter pack extend 0.9 to 1.5 m (3 to 5 ft) above the screen? Y__N__N/A__
11. Is bentonite seal to be adequately hydrated or fine sand placed to prevent grout intrusion? Y__N__N/A__
12. Is grout placed appropriately and to the proper level? Y__N__N/A__
13. Are the wells adequately protected? Y__N__N/A__
14. Has possible damage from frost heave been considered in the well design? Y__N__N/A__
15. Do aboveground installations have a drainhole near the base of the protective casing? Y__N__N/A__
16. Does well have a lockable well cap for security? Y__N__N/A__
17. Is the concrete/gravel pad around the well adequate? Y__N__N/A__
18. Are the well coordinates and elevations surveyed? Y__N__N/A__
19. Will wells be developed by bailing and purging? Y__N__N/A__
20. Is well development record maintained? Y__N__N/A__
21. Will field measurements of the groundwater be taken? Y__N__N/A__
22. Are soil borings properly backfilled/abandoned? Y__N__N/A__
23. Will soil borings be logged by a geologist-geotechnical engineer? Y__N__N/A__
24. Are logging procedures discussed? Y__N__N/A__
25. Are rock cores logged and photographed? Y__N__N/A__
26. Is disposal of soil cuttings, well development water, decontamination water, and other wastes addressed? Y__N__N/A__

27. Is a sample boring log with a scale provided? Y__N__N/A__
28. Is a list of field equipment provided? Y__N__N/A__

29. Are sample well installation diagram and development record form provided? Y__N__N/A__
30. Are all standard field parameters to be recorded? Y__N__N/A__
31. Is a hard-bound logbook maintained? Y__N__N/A__
32. Are slug test procedures described? Y__N__N/A__

c. Groundwater Sampling
1. Are water level measurements taken before well purging? Y__N__N/A__
2. Are 3 to 5 well volumes purged prior to sampling the well? Y__N__N/A__
3. Are sampling devices described? Y__N__N/A__
4. Are purging devices described? Y__N__N/A__
5. Is filtration method described for collecting sample for dissolved metals? Y__N__N/A__
6. Are methods to obtain field measurements (pH, temperature, specific conductivity) described? Y__N__N/A__
7. Are sampling devices decontaminated between samples? Y__N__N/A__
8. Are procedures for collecting QA/QC samples addressed? Y__N__N/A__
9. Are trip blanks sent with samples for volatile organic analysis? Y__N__N/A__

d. Soil Sampling
1. Is sampling equipment described and appropriate for the purpose and site conditions? Y__N__N/A__
2. Are sample containers for volatiles filled before soil is composited? Y__N__N/A__
3. Is head space in sample containers for volatiles eliminated? Y__N__N/A__
4. Is sampling instrument decontaminated between samples? Y__N__N/A__
5. Are procedures for collecting QA/QC samples addressed? Y__N__N/A__
6. Are trip blanks sent with samples for volatile organic analysis? Y__N__N/A__

e. Sediment Sampling
1. Are sample locations referenced to a permanent structure and located with field measurements? Y__N__N/A__
2. Are sediment samples collected after surface water samples? Y__N__N/A__
3. Are sampling instruments appropriate? Y__N__N/A__
4. Are sampling instruments decontaminated between samples? Y__N__N/A__
5. Are excess water, sticks, rocks, and other debris removed before placing sediment into sample containers? Y__N__N/A__
6. Are procedures for collecting QA/QC samples addressed? Y__N__N/A__

f. Surface Water Sampling
1. Is surface water sample collected before sediment sample? Y__N__N/A__
2. Is depth of water measured? Y__N__N/A__
3. Are sampling instruments described and appropriate for purpose and site conditions? Y__N__N/A__
4. Are sampling procedures described? Y__N__N/A__
5. Are methods to obtain field measurements (pH, temperature, specific conductance) described? Y__N__N/A__
6. Are sampling instruments decontaminated between samples? Y__N__N/A__
7. Are procedures for collecting QA/QC samples described? Y__N__N/A__

g. Sample Packaging and Shipping
1. Are samples required to be chilled immediately after

being collected? Y__N__N/A__
2. Are shipping coolers made of suitable material? Y__N__N/A__
3. Is empty space in cooler filled with insert packing material? Y__N__N/A__
4. Are bottles enclosed in clean plastic bags? Y__N__N/A__
5. Are sample tags affixed to sample containers? Y__N__N/A__
6. Are bottles placed upright in cooler in a way that they do not touch? Y__N__N/A__
7. Are bags of ice placed in coolers containing samples for chemical analysis? Y__N__N/A__
8. Is chain of custody form sealed in plastic bag and taped to inside lid of cooler? Y__N__N/A__
9. Is cooler drain taped shut? Y__N__N/A__
10. Is cooler lid secured with tape? Y__N__N/A__
11. Is completed shipping label taped to top of cooler? Y__N__N/A__
12. Are "This Side Up" labels placed on all four sides of cooler? Y__N__N/A__
13. Are "fragile" labels placed on two sides of coolers? Y__N__N/A__
14. Are signed custody seals affixed to the front right and left side of the coolers? Y__N__N/A__
15. Are medium/high concentration samples placed in metal cans and secured with three clips prior to placement in cooler? Y__N__N/A__
16. Are metal cans containing medium/high concentration samples properly labeled? Y__N__N/A__

h. Is a schedule for the field activities presented? Y__N__N/A__

i. Are daily quality control reports described? Y__N__N/A__
   1. Are notification and corrective action procedures discussed? Y__N__N/A__
   2. Are procedures to deviate from approved SAP described? Y__N__N/A__

j. Is disposal of RI-derived wastes properly documented? Y__N__N/A__

QUALITY ASSURANCE PROJECT PLAN (QAPP)

Quality Assurance Objectives
(This information may be referenced to the Project Work Plan.)
a. Are field measurement objectives discussed? Y__N__N/A__
b. Are analytical method detection limits defined? Y__N__N/A__
c. Are quality control parameters defined? Y__N__N/A__
   1. Precision and accuracy Y__N__N/A__
   2. Completeness Y__N__N/A__
   3. Representativeness Y__N__N/A__
   4. Comparability Y__N__N/A__

Sample Custody/Documentation
a. Is a field logbook maintained with appropriate information concerning drilling/sampling? Y__N__N/A__
b. Is method of identifying photographs discussed? Y__N__N/A__
c. Is sample numbering system appropriate? Y__N__N/A__
   1. Project designator Y__N__N/A__
   2. Location designation Y__N__N/A__
   3. Matrix code Y__N__N/A__
   4. Sample sequence numbers Y__N__N/A__
   5. Depth interval (if required) Y__N__N/A__

d. Sample Documentation
   1. Does information on sample label include:
   -- Site name Y__N__N/A__

-- Identification of sample station number	Y__N__N/A__
-- Date and time of collection	Y__N__N/A__
-- Name of sampler	Y__N__N/A__
-- Analytical analyses requested	Y__N__N/A__
-- Media sampled	Y__N__N/A__
-- Preservation method	Y__N__N/A__
2. Are completed custody seals required over sample container (except VOA) lids?	Y__N__N/A__
3. Does chain-of-custody record contain appropriate information?	Y__N__N/A__
4. Are receipts for sample forms required?	Y__N__N/A__
5. Are the step-by-step sample documentation procedures explained?	Y__N__N/A__
6. Are procedures to correct sample documentation explained?	Y__N__N/A__

Laboratory Analytical Procedures

a. Is laboratory QA plan available?	Y__N__N/A__
b. Are analytical methods specified?	Y__N__N/A__
c. Are detection limits specified?	Y__N__N/A__
d. Are performance and systems audits described and scheduled?	Y__N__N/A__
e. Is preventive maintenance addressed?	Y__N__N/A__
f. Are instrument calibration procedures and frequency addressed?	Y__N__N/A__
g. Are laboratory's data reduction, validation, and documentation and custody procedures addressed?	Y__N__N/A__
h. Are requirements for timing of data submittals, reporting format and contents, and recipients of data addressed?	Y__N__N/A__

CONCLUSION

_____ Approval Recommended

_____ Approval Recommended with Comments

_____ Resubmission Recommended

Reviewed: _____

Date: _____

Glossary

Acronyms

AA	Atomic absorption
AASHTO	American Association of State Highway and Transportation Officials
ACGIH	American Conference of Governmental Industrial Hygienists
ACE	U.S. Army Corps of Engineers (EPA terminology)
ACE	Assistant Chief of Engineers
ACS	American Chemical Society
A-E	Architect - Engineer
AES	Atomic emission spectroscopy
AF	Air Force
AFB	Air Force Base
AL	Action level
ALARA	As low as reasonably achievable
amu	Atomic mass units
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
AOC	Area of concern
APA	Air pathway analysis
APC	Air pollution control
ARAR	Applicable, or relevant and appropriate requirements
ASAP	Adaptive Sampling and Analysis Plan
ASE	Accelerated solvent extraction
ASQC	American Society for Quality Control
AST	Aboveground storage tank
ASTM	American Society for Testing and Materials
ATSDR	Agency for Toxic Substances and Disease Registry
AVO	Aromatic volatile organics
AWQC	Ambient water quality criteria
BACT	Best available control technology
BAT	Best available technology
BDAT	Best demonstrated available technology
BFB	Bromofluorobenzene
BIF	Boilers and industrial furnaces
BNA	Base, neutral, acids (semivolatile organics)
BNA	Bureau of National Affairs
BOD	Biological oxygen demand
BOE	Bureau of Explosives
BP	Boiling point
BRA	Baseline risk assessment
BRAC	Base Realignment and Closure
BTEX	Benzene, toluene, ethylbenzene, and xylene
CA	Corrective action
CAA	Clean Air Act
CAAA	Clean Air Act amendments
CADD	Computer aided design and drafting
CAMU	Corrective action management unit

CAS	Chemical Abstract Service
CATV	Corrective action treatment unit
CBD	Commerce Business Daily
CCB	Continuing calibration blank
CCC	Calibration check compound
CCQC	Contractor Chemical Quality Control
CCV	Continuing calibration verification
CD	Consent decree
CDAP	Chemical Data Acquisition Plan
CDC	Centers for Disease Control
CDQAR	Contractor Data Quality Assessment Report
CDQM	Chemical data quality management
CEFMS	Corps of Engineers Financial Management System
CEGS	U.S. Army Corps of Engineers Guide Specification
CEM	Continuous emission monitors
CERCLA	Comprehensive Environmental Response, Compensation, Liability Act
CERCLIS	CERCLA Information System
CF	Calibration factor
CFC	Chlorofluorocarbon
CFR	Code of Federal Regulations
CGM	Combustible gas meter
CHMM	Certified Hazardous Material Manager
CL	Confidence level
CLP	Contract Laboratory Program
CMA	Chemical Manufacturers Association
CME	Central mine equipment (sampler)
CMECC	California Military Environmental Coordination Committee
CMI	Corrective Measures Implementation
CMS	Corrective Measures Studies
CNAEL	Committee on National Accreditation of Environmental Laboratories
CO	Contracting Officer
COC	Chain of custody
COC	Contaminants of concern
COD	Chemical oxygen demand
COE	U.S. Army Corps of Engineers
COELT	U.S. Army Corps of Engineers loading tool
COLIWASA	Composite liquid waste sampler
CPT	Cone penetrometer testing
CQAR	Chemical Quality Assurance Report
CQC	Contractor quality control
CRADA	Cooperative research and development agreements
CRDL	Contractor-required detection limit
CRP	Community Relations Plan
CRQL	Contractor-required quantitation limit
CRREL	Cold Regions Research and Environmental Laboratory
CRT	Cathode ray tube
CSCT	Consortium for Site Characterization Technologies
CSM	Conceptual site model
CSR	Constant sampling rate

CV (COV)	Coefficient of variation
CVAA	Cold vapor atomic absorption
CWA	Clean Water Act
CX	Center of Expertise
DCB	Decachlorobiphenyl
DCQAP	Data Collection Quality Assurance Plan
DDD	Dichlorodiphenyldichloroethane
DDE	Dichlorodiphenyldichloroethylene
DDT	Dichlorodiphenyltrichloroethane
DEEMS	Department of Energy Environmental Management Electronic Data Deliverable Master Specification
DEIS	Draft Environmental Impact Statement
DERA	Defense Environmental Restoration Account
DERP	Defense Environmental Restoration Program
DFTPP	Decafluorotriphenylphosphate
DL	Detection limit
DMP	Data Management Plan
DNAPL	Dense non-aqueous phase liquid
DNB	Dinitrobenzene
DNT	Dinitrotoluene
DO	Dissolved oxygen
DOD	Department of Defense
DOI	Department of Interior
DOE	Department of Energy
DOT	Department of Transportation
DPM	Defense priority model
DQI	Data quality indicator
DQO	Data quality objective
DRE	Destruction and removal efficiency
DTW	Depth to water
DWPL	Drinking Water Priority List
EA	Endangerment assessment
EB	Equipment blank
ECD	Electron capture detector
EDF	Environmental Defense Fund
EDD	Electronic data deliverable
EE/CA	Engineering evaluation/cost analysis
EHS	Extremely hazardous substances
EHW	Extremely hazardous waste
EIA	Enzyme immunoassay
EICP	Extracted ion current profile
EIR	Environmental Impact Report
EIS	Environmental Impact Statement
ELCD	Electrolytic conductivity detector
EM	Engineer manual
EMC	Emission Measurement Center
EO	Executive Order
EO	Explosive ordnance
EOD	Explosive ordnance disposal

EP	Engineer pamphlet
EP Tox	Extraction procedure toxicity
EPA	U.S. Environmental Protection Agency
EQL	Estimated quantitation limit
ER	Engineer regulation
ESC	Expedited Site Characterization
ESTCP	Environmental Security Technology Certification Program
ETL	Engineer technical letter
eV	Electron volt
FAR	Field analytical result
FAR	Federal Acquisition Regulation
FDE	Findings and determination of eligibility
FEMA	Federal Emergency Management Agency
FFA	Federal facility agreement
FFMS	Fixed-fenceline measurement system
FFP	Firm fixed price
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FID	Flame ionization detector
FLAA	Flame atomic absorption
FN	False negative
FP	False positive
FP	Flashpoint
FR	Federal Register
FRTR	Federal Remediation Technologies Roundtable
FS	Feasibility study
FSP	Field Sampling Plan
FTIR	Fourier transformed infrared (spectroscopy)
FUDS	Formerly used defense site
FY	Fiscal year
GAC	Granulated activated carbon
GALP	Good automated laboratory practices
GAO	Government Accounting Office
GC	Gas chromatograph or gas chromatography
GC/MS	Gas chromatograph/mass spectrometer
GFAA	Graphite furnace atomic absorption
GIS	Geographic Information System
GLP	Good laboratory practices
GPC	Gel permeation column (chromatography)
GPM	Gallons per minute
GPR	Ground penetrating radar
HAP	Hazardous air pollutant
HAZCAT	Hazardous characterization (testing)
HAZMAT	Hazardous materials
HAZWRAP	Hazardous Waste Remedial Program
HDPE	High density polyethylene
HE	High explosive
HECD	Hall electrolytic conductivity detector
HM	Hazardous material
HMX	Cyclotetramethylenetetranitramine (Her majesty's explosive)

HPLC	High performance liquid chromatography
HQUSACE	Headquarters, U.S. Army Corps of Engineers
HRGC	High resolution gas chromatography
HRMS	High resolution mass spectrometry
HRS	Hazard ranking system
HSL	Hazardous substance list (TAL + TCL)
HSWA	Hazardous and solid waste amendments
HTRW	Hazardous, toxic, and radioactive waste
HTRW-CX	Hazardous, Toxic, and Radioactive Waste - Center of Expertise
HVO	Halogenated volatile organics
IAG	Interagency agreement
IATA	International Air Transportation Association
IC	Ion chromatography
ICAO	International Civil Aviation Organization
ICAP	Inductively coupled argon plasma emission spectroscopy
ICB	Initial calibration blank
ICP	Inductively coupled plasma
ICP/MS	Inductively coupled plasma / mass spectrometer
ICS	Interference check standard
ICV	Initial calibration verification
ID	Identification
IDL	Instrument detection limit
IDW	Investigation-derived waste
IEC	Interelement correction standard
IEC	International Electrotechnical Commission
INPR	Inventory project report
IPR	Inventory project report
IR	Infrared radiation
IRP	Installation Restoration Program
IRPMIS	Installation Restoration Program Management Information System
IRIS	Integrated Risk Information System
ISE	Ion selective electrode
ISO	International Standards Organization
ITA	Innovative technology advocate
ITRC	Interstate Technology Regulatory Cooperation
IUPAC	International Union of Pure and Applied Chemistry
K-D	Kuderna-Danish
LAER	Lowest achievable emissions rate
LC	Liquid chromatography
LCL	Lower control limit
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LDR	Land disposal restrictions (LANDBAN)
LDPE	Low density polyethylene
LFG	Landfill gas
LIBS	Lasar-induced breakdown spectroscopy
LIF	Lasar-induced fluorescence
LNAPL	Light, non-aqueous phase liquid
LNC	Laboratory notification checklist

LIMS	Laboratory information management system
LLE	Liquid-liquid extraction
LLW	Low level waste (radioactive)
LOD	Limit of detection
LOQ	Limit of quantitation
LQMP	Laboratory Quality Management Plan
LSE	Liquid-solid extraction
LUST	Leaking underground storage tank
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MB	Method blank
MCAWW	Methods for Chemical Analysis of Water and Wastes
MCL	Maximum contaminant level
MCLG	Maximum contaminant level goal
MCS	Media cleanup standards
MD	Matrix duplicate
MDL	Method detection limit
MDRD	Minimum detectable relative difference
MEK	Methyl ethyl ketone (2-butanone)
MFR	Memorandum for record
mg/Kg	Milligram per kilogram
MOA	Memorandum of agreement
MOU	Memorandum of understanding
MPS	Multi-port sampler
MQL	Method quantitation limit
MQO	Measurement quality objective
MRL	Method reporting limit
MS	Mass spectrometer
MS	Matrix spike
MSA	Method of standard additions
MSC	Major subordinate commands
MSD	Matrix spike duplicate
MSDS	Material safety data sheet
MSL	Mean sea level
MSW	Municipal solid waste
MW	Molecular weight
MW	Monitoring well
MWIP	Monitoring well installation plan
NA	North America
NA	Not applicable
NAAQS	National ambient air quality standards
NAPL	Non-aqueous phase liquid
NAS	Network analysis system
NBS	National Bureau of Standards
NCP	National Contingency Plan
NERL-LV	EPA National Environmental Research Laboratory - Las Vegas
ND	Non-detect
NEIC	National Enforcement Investigations Center (EPA)
NEPA	National Environmental Policy Act
NESHAP	National Emission Standards for Hazardous Air Pollutants

NFA	No further action
NHPA	National Historic Preservation Act
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology (formerly NBS)
NOI	Notice of intent
NOIBN	Not otherwise indicated by name
NOS	Not otherwise specified
NOV	Notice of Violation
NPD	Nitrogen - phosphorus detector
NPDES	National Pollutant Discharge Elimination System
NPDWR	National Pollution Drinking Water Regulation
NPL	National Priorities List
NPS	Non-point source
NRC	Nuclear Regulatory Commission
NRC	National Response Center
NSPS	National source performance standards
NT	Nitrotoluene
NTU	Nephelometric turbidity unit
NWS	National weather station (service)
OAC	Other areas of concern
O & M	Operations and maintenance
OB/OD	Open burning / open detonation
OCE	Office of Chief of Engineers
OERR	EPA Office of Emergency and Remedial Response
OEW	Ordnance and explosive waste
OMB	Office of Management and Budget
OMSQA	Office of Monitoring Systems and Quality Assurance
ORP	Oxidation-reduction potential
OSC	On-scene coordinator
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
OTA	Office of Technology and Assessment
OU	Operable unit
OVA	Organic vapor analyzer
PA	Performance audit
PA/SI	Preliminary Assessment/Site Inspection
PAC	Powdered activated carbon
PAH	Polynuclear aromatic hydrocarbon
PARCCS	Precision, accuracy, representativeness, comparability, completeness, and sensitivity
PAT	Proficiency analytical testing
PAWS	Portable acoustic wave sensor system
PB	Preparation blank
PBMS	Performance based measurement system
PC	Polycarbonate
PCB	Polychlorinated biphenyl
PCP	Pentachlorophenol
PDS	Post digestion spike

PE	Performance evaluation
PE	Professional engineer
PF	Protection factor
PG	Professional geologist
PG	Packing group
PID	Photoionization detector
PM	Particulate matter
PM	Project manager
PNA	Polynuclear aromatic (hydrocarbons)
POC	Purgeable organic carbon
POC	Point of contact
POHC	Principal organic hazardous constituent
POL	Petroleum, oils, and lubricants
POTW	Publicly-owned treatment works
POX	Purgeable organic halides
ppb	Parts per billion (e.g., • g/L or • g/Kg)
PPE	Personal protection equipment
ppm	Parts per million (e.g., mg/L or mg/Kg)
ppt	Parts per trillion
PRG	Preliminary remediation goal
PQL	Practical quantitation limit
PRP	Potentially responsible party
PSI	Per square inch
PSN	Proper shipping name
PSR	Particle size reduction
PSS	Particle size separation
PT	Purge and trap
PTFE	Polytetrafluoroethylene
PUF	Poly urethane foam
PVC	Polyvinyl chloride
QA	Quality assurance
QAMS	Quality assurance management systems
QAMS	Quality assurance management staff
QAPP	Quality Assurance Project Plan
QC	Quality control
QCR	Quality Control Report
QCSR	Quality Control Summary Report
QL	Quantitation limit
RA	Remedial action
RAS	Routine analytical services
RC	Remedial construction
RCRA	Resource Conservation Recovery Act
RD	Remedial design
RDX	Cyclo-1,3,5-trimethylene-2,4,6-trinitramine (Royal demolition explosive)
RF	Response factor
RFA	RCRA Facility Assessment
RFI	RCRA Facility Investigation
RI/FS	Remedial Investigation/Feasibility Study

RL	Reporting limit
RMCL	Recommended maximum contaminant level
ROD	Record of decision
ROE	Right of entry
RPD	Relative percent difference
RPM	Remedial project manager
RQ	Reportable quantities
RRT	Relative retention time
RSD	Relative standard deviation
RSE	Relative standard error
S & A	Supervision and administration
SACM	Superfund Accelerated Cleanup Model
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SAS	Special analytical services
SAW	Surface acoustic wave array detector
SCAPS	Site characterization and analysis penotrometer system
SD	Standard deviation
SD	Serial dilution
SDWA	Safe Drinking Water Act
SGS	Soil gas survey
SI	Site investigation
SIP	State Implementation Plan
SITE	Superfund innovative technology evaluation
SMCL	Secondary maximum contaminant level
SMF	Sporadic marginal failure
SOC	Synthetic organic compound
SOP	Standard operating procedures
SOW	Scope of work
SPCC	System performance check compound
SPMD	Semi-permeable membrane device
SPE	Solid phase extraction
SPP	Sample preparation procedure
SQG	Small quantity generator
SQL	Sample quantitation limit
SRM	Standard reference material
SSHP	Site safety and health plan
SV	Sampling visit
SVE	Soil vapor extraction
SVOC	Semivolatile organic compound
SW-846	Test Methods for Evaluating Solid Waste-Physical/Chemical Methods analytical protocols
SWDA	Solid Waste Disposal Act
SWMU	Solid Waste Management Unit
TAL	Target analyte list (CLP inorganics)
TAP	Toxic air pollutant
TAT	Technical assistance team
TB	Trip blank
TBC	To be considered
TBT	Tributyl tin

TCDD	Tetrachlorodibenzodioxin
TCDF	Tetrachlorodibenzofuran
TCE	Trichloroethylene
TCL	Target compound list (CLP organics)
TCLP	Toxicity characteristic leaching procedure
TDS	Total dissolved solids
TERC	Total environmental restoration contract
THM	Trihalomethane
TIC	Tentatively identified compound
TIO	EPA Technology Innovation Office
TLC	Thin layer chromatography
TM	Technical manager
TNT	Trinitrotoluene
TO	Toxic organics
TOC	Total organic carbon
TOX	Total organic halides
TPH	Total petroleum hydrocarbons
TPP	Technical project planning
TRPH	Total recoverable petroleum hydrocarbons
TSCA	Toxic Substances Control Act
TSD	Treatment, storage, disposal (facility)
TSP	Total suspended particulates
TSS	Total suspended solids
TSWP	Treatability Study Work Plan
TTN	Technology transfer network
UIC	Underground injection control
UCL	Upper control limit
UN	United Nations
USACE	U.S. Army Corps of Engineers
USCG	U.S. Coast Guard
USCS	Unified Soil Classification System
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
UST	Underground storage tank
UV	Ultraviolet
UXO	Unexploded explosive ordnance
VES	Vapor Extraction System
VOA	Volatile organic analysis (analyte)
VOC	Volatile organic compounds
VP	Vapor pressure
VSI	Visual site inspection
WP	White phosphorus
WQC	Water quality criteria
XRF	X-ray fluorescence
ZHE	Zero headspace extractor (TCLP VOCs)

Terms

Accuracy: the closeness of agreement between the measured value and the true value. Calculated as percent recovery.

Activity: an all-inclusive term describing a set of operations or related tasks to be performed, either serially or in parallel, that result in a total product or service.

Aliquot: a measured portion of a sample taken for analysis (USEPA CLP Statement of Work).

Analyte: a discrete chemical component of a sample to be identified and/or measured through analysis.

Anion: a negatively charged ion.

Aquifer: a geologic formation, group of formations, or part of a formation capable of yielding a significant amount of groundwater to wells or springs.

Aromatic: relating to the six-carbon-ring configuration of benzene and its derivatives.

Audit: an independent, systematic examination to determine whether activities are effective and comply with planned arrangements, and whether the results are suitable to achieve objectives.

Background concentrations or levels: average presence in the environment (USEPA). Concentrations of contaminants detected in environmental samples from various media on the site or in the area of the site that have not been affected by site operations. These concentrations may reflect the natural occurrence of elements, as in the case of metals in soil. They may also reflect the widespread presence of compounds resulting from a variety of industrial and commercial activities, as in the case of PAHs in surface soils in urban areas.

- Regional background concentrations--usually apply to soil and reference data from a resource such as Shacklette and Boerngen (1984).

- Site-specific background concentrations--reference actual samples collected on the site or in the area of the site. Examples of such samples are ground water samples from a monitoring well upgradient of the site or surface soil samples from an area that has not been affected by onsite operations.

Bar Graph Spectrum: a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

Batch (preparatory): batch is the basic unit for quality control implementation. The batch is defined as a group of • 20, similar matrix samples and all of the required quality control samples that are analyzed together following the same method sequence, with the same manipulations, using the same reagents, during the same time period.

Bias: the systemic or persistent distortion of a measurement process that causes errors in one direction.

Boring: a cylindrical hole advanced into the ground, usually made by drilling.

Bottle Blank: analyte-free (deionized) water transferred to the appropriate sample bottles in the field and submitted for analysis. Results assess the potential incidental (airborne) contamination and cross-contamination due to the sample bottles and preservatives.

4-Bromofluorobenzene (BFB): a compound chosen to establish mass spectral instrument performance for volatile analysis.

Calibration: determination of the ratio of instrument response to analyte concentration. Established by the analysis of standards containing analytes of interest at known concentrations.

Calibration Check Compounds (CCC): term used in conjunction with Method 8260 (EPA/SW-846) to refer to the compounds in which the percent relative standard deviation is evaluated against method-prescribed criteria to decide the validity of a calibration.

Calibration Standards (CAL): a set of solutions prepared from the primary standards solution with the internal standards and surrogate analytes as appropriate, used to calibrate the instrument response with respect to analyte concentration.

Cation: a positively charged ion.

CERCLA/SARA: the Comprehensive Environmental Response, Compensation, and Liability Act of 1980, as amended by the Superfund Amendments and Reauthorization Act of 1986. The acronym CERCLA is frequently used to refer to both acts, as is the term Superfund. CERCLA requires the administrator of the USEPA to promulgate regulations (see NCP) designating hazardous substances that, when released into the environment, may present substantial danger to public health, welfare, or the environment. The act established the Superfund and required the promulgation of regulations governing the funding and cleanup of waste sites and contaminated areas. CERCLA is the act that establishes legislative authority, while the National Oil and Hazardous Substances Contingency Plan (NCP) is the regulation that implements the requirements of CERCLA.

Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples, data, and records.

Characteristic: any property or attribute of a datum, item, process, or service that is distinct, describable, or measurable.

Chemical Analysis: any of a variety of laboratory methods used to evaluate the concentrations of compounds and elements present in an environmental sample.

Cleanup Goals/Cleanup Standards/Cleanup Levels/Cleanup Criteria/Remediation Goals/Action Levels: for consistency, the following program usage is suggested:

- Action levels--to refer to the presence of a contaminant concentration in the environment high enough to warrant action or trigger a response under CERCLA or the National Oil and Hazardous Substances Contingency Plan (USEPA).

Cleanup/Remediation/Remedial Action/Removal/Removal Action: for consistency, the following program usage is suggested:

- Remedial action--refers to all activities, except long-term operation and maintenance, associated with permanent correction or remedy of contamination at or in the area of a site.

- Removal action--refers to limited or short-term measures intended to mitigate the immediate effects of or prevent the release of hazardous substances into the environment (specifically, source removal).

- Cleanup or remediation--refers to all activities, including long-term operation and maintenance, associated with permanent correction or remedy of contamination at or in the area of a site.

Comparability: a qualitative characteristic that defines the extent to which the data for a chemical parameter measurement are consistent with, and may be compared with, data from other sampling events.

Compatibility: the ability of materials (and/or wastes) to coexist without adverse effects.

Completeness: a quantitative evaluation of what percent of the chemical measurements (results) are successfully accomplished.

Composite Sample: portions of material collected from more than one spatial location or at different times that are blended and submitted for chemical analyses. Composite samples can provide data representative of a large area with relatively few samples. However, the resulting data are less accurate with regard to the concentrations of contaminants detected in a specific location, because they represent average values.

Compound: a substance composed of two or more elements existing in combination. Each compound may be expressed by a chemical formula.

Continuing Calibration Verification Standard (CCV): a midconcentration analytical standard run periodically to verify the calibration of the analytical instrument is valid.

Continuous Barrel Sampler: a 1.5-m- (5-ft-) long split barrel sampler used to collect representative samples of soil or soft rock. The sampler consists of five parts: a cutting shoe at the bottom, a barrel consisting of a length of pipe split longitudinally into two halves, a sample catcher, and a coupling at the top for connection to the drill rods.

Contract Laboratory Program (CLP): a nationwide laboratory network established by the USEPA, structured to provide legally defensible analytical results to support USEPA enforcement actions or other requirements of the user community. The CLP incorporates a level of quality assurance appropriately designed for the intended usage of the data.

Contractor Chemical Quality Control: a three-phase control process (preparatory, initial, and follow-up) that is performed onsite by the contractor to ensure that quality is maintained throughout all field work.

Corrective Action: measures taken to rectify conditions adverse to quality and where possible, to preclude their recurrence.

Data Assessment: the all-inclusive process used to measure the effectiveness of a particular data-gathering activity. This process may comprise data verification, data review, data evaluation, and data validation.

Data Evaluation: The process of data assessment done by USACE District project chemist to produce a Contractor Data Quality Assessment Report. Refer to EM 200-1-6.

Data Quality Indicators (DQI): measurable attributes for the attainment of necessary quality to support an environmental decision. DQIs include precision, bias, completeness, representativeness, reproducibility, comparability, sensitivity, and statistical confidence.

Data Quality Objectives: qualitative and quantitative statements that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for decisions.

Data Review: an evaluation of laboratory data quality based on a review of method-specific quality control documentation. Method-specific quality control documentation requirements are specified in the project-specific laboratory subcontract.

Data Validation: an evaluation of laboratory data quality based on a review of the data deliverables. This process involves procedures verifying instrument calibration, calibration verification, and other method-specific performance criterion.

Data Verification: the process for evaluating the completeness, correctness, consistency, and compliance of a data package against a standard or contract.

Decontamination: cleaning of personnel, equipment, structural materials, etc., using any of a variety of technologies. The most commonly used technologies are washing, using soap and water and/or various acidic rinses or solvents, etc.; and steam cleaning. This term applies both to cleaning of personnel and equipment following site investigation and remediation activities and to cleaning of contaminated structures or structural materials as part of a removal or remedial action.

Deficiency: an unauthorized deviation from approved procedures or practices, or a defect in an item.

Definitive Data: data that are generated using rigorous, analyte-specific analytical methods where analyte identification and quantitations are confirmed and quality assurance/quality control requirements are satisfied.

Detection Limit: the minimum concentration of an analyte that can be measured within a given matrix and reported with a 99 percent confidence that the analyte concentration is greater than zero.

Discrete Sample: a portion of material collected from a unique spatial location and submitted for chemical analyses. Discrete samples are collected when it is necessary to identify and quantify contamination at a specific location and time.

Disposal: final placement or destruction of wastes. Disposal may be accomplished through the use of landfills, treatment processes, etc.

Dissolved Metals: the concentration of metals determined in a sample that will pass through a 0.45-μm or appropriately sized filter. The sample is filtered, and the filtrate is preserved (acidified) in the field, transported to the laboratory, and then analyzed following appropriate methodologies.

Duplicate: see Matrix Duplicate.

Environmental Sampling: collection of samples from a particular media for the purpose of obtaining chemical analyses.

Equipment Rinsate Blank/Field Equipment Blank/Rinsate Blank/Equipment Blank: samples of clean, analyte-free water passed through and over the sampling equipment. These blanks permit evaluation of equipment decontamination procedures and potential cross-contamination of environmental samples between sampling locations. An equipment rinsate blank is typically obtained from each type of sampling tool used to collect environmental samples.

Extractable Organics: semivolatiles (base/neutral and acid-extractable compounds) and pesticide/ polychlorinated biphenyl compounds that can be partitioned into an organic solvent from the sample matrix and are amenable to gas chromatography.

Feasibility Study (FS): a description and analysis of the potential cleanup alternatives for a hazardous waste site. Cleanup alternatives are broadly evaluated on the basis of effectiveness, implementability, and cost. The USEPA "Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA" specifies nine detailed evaluation criteria (EPA/540/G-89/004).

Field Blank: a general term used to describe various blanks, including bottle blanks, equipment blanks, media blanks, trip blanks, etc.

Field Control Samples: general term assigned to field-generated quality assurance/quality control samples, such as replicates (duplicates/splits/spikes), blanks, background/upgradient samples, etc.

Field Duplicate Sample: independent sample collected at approximately the same time and place, using the same methods as another sample. The duplicate and original samples are containerized, handled, and analyzed in an identical manner.

Field Investigation: any investigation conducted at a site or in the area of a site for the purpose of site characterization. A field investigation may or may not be part of a remedial investigation or remedial action. It may include geophysical surveys, ground surveys, well surveys, environmental sampling, etc.

Field Replicate: a general term for field duplicates/triplicates, field splits, or field spikes. Samples may be homogenized prior to splitting into replicate samples. Each replicate is containerized, handled, and analyzed in an identical manner. Used to evaluate the precision of handling, shipping, storage, preparation, and analysis.

Filtrate: a filtered liquid.

Filtration: the physical removal of solid particles from a liquid waste stream by passing the liquid across a filter medium, which serves as a barrier to the solid material.

Field Sampling Plan (FSP): the portion of the Sampling Analysis Plan that defines the field activities; includes all requirements for sampling, field documentation, onsite chemical analysis, sample packaging and shipping, etc.

Gas Chromatography: A process in which the components of a mixture are separated from one another by volatilizing the sample mixture into a carrier gas stream that is passing through and over a bed of packing solid support. Different components move through at different rates depending on their size and affinity toward the solid support. Elution from the column occurs at different rates to the various detectors where the components are measured based on thermal conductivity changes, density differences, or ionization

detectors. The primary components of a gas chromatograph include injection port, column, integrator/data system, and detectors.

Gas Chromatography/Mass Spectroscopy (GC/MS): two distinct analytical techniques used to separate and identify organic compounds: the GC is used for the separating portion and the MS is used as the detection portion of an analysis. Both techniques are typically performed by a single instrument.

Grab Sample: an individual sample collected from a single location at a specific time. Samples are collected and placed in the appropriate sample containers with no mixing.

Hazardous/Nonhazardous: the following terms are correct:

- Hazardous waste (RCRA)--as defined in 40 CFR 261, byproducts of society that can pose a substantial or potential hazard to human health or the environment when improperly managed. Refers to both wastes listed in the referenced section and wastes demonstrating any of the four hazardous characteristics (ignitability, corrosivity, reactivity, and toxicity) identified in the referenced section.

- Hazardous substance (CERCLA)--encompasses not only RCRA hazardous wastes, but also includes substances and pollutants listed under the Clean Water Act; hazardous air pollutants listed under the Clean Air Act; any substance with respect to which the USEPA has taken action under the Toxic Substances Control Act; and elements, compounds, mixtures, solutions, and substances (to be identified by the USEPA under CERCLA) which, when released into the environment, may present substantial danger to the public welfare or the environment.

- Hazardous material (Department of Transportation)--refers to materials contaminated by any substance that is listed in the appendix to 49 CFR 172.101 and that exceeds the reportable quantity criteria identified in this appendix.

- Nonhazardous--if used, clarify whether it is used as the opposite of one or all of these terms, or whether it refers to the absence of toxic characteristics as defined by risk assessment techniques, etc.

Heavy Metals: in reference to environmental sampling, typically identified as the following trace inorganics: cadmium, lead, mercury, silver, etc. (all metals of health concern). Heavy metals can cause biological damage if consumed at low concentrations and tend to accumulate in the food chain.

Heterogeneous: the quality of containing dissimilar parts within the composition of the media.

High-Performance Liquid Chromatography (HPLC): an analytical technique used for separating and identifying compounds not amenable to gas chromatography.

Homogeneous: the quality of uniform composition.

Homogenized Sample: a sample collected from a single location at a specific time, but mixed to ensure representativeness prior to containerizing. This technique is not suitable for volatile organic samples.

Hydrogeologic Investigation: a systematic study of the interrelationships that exist between geology and the associated ground and surface water.

Hydrogeology: the study of the interrelationships of geologic materials and processes with water, especially groundwater.

Hydrology: the study of the occurrence, distribution, and chemistry of all waters of the earth.

Infiltration: the penetration of water through the ground surface into subsurface soil or the penetration of water from the soil into sewer or other pipes through defective joints, connections, or manhole walls.

Initial (Continuing) Calibration Blank (ICB/CCB): a volume of ASTM D 1193 Type II (polished) water prepared in the same manner as standards used to flush the analytical system.

Initial (Continuing) Calibration Verification Standard (also instrument check standard) (ICV/CCV): a USEPA-certified multielement standard or independently prepared multielement standard solution used to verify the accuracy of the initial calibration. This standard prepares all elements at solutions of known concentrations equivalent to the midpoint of their respective calibration curves and must be run at each wavelength used in the inductively coupled plasma analysis.

Inorganic Chemicals: chemical substances of mineral origin, not of basically carbon structure.

Interference (Interelement) Check Standard (ICS or IEC): a solution containing both interfering and analyte elements of known concentrations used to verify background and/or interelement interferences, so that appropriate correction factors are utilized to compensate.

Internal Standards (IS): Compounds added to every standard, blank, sample, matrix duplicate, matrix spike, matrix spike duplicate, etc., at a known concentration, prior to analysis by GC or GC/MS when using internal standard calibration and quantitation techniques. Internal standards are used as the basis for quantitation of the target compounds.

Laboratory Control Sample (LCS): also referred to as a QC (Reference) Sample. A spiked blank sample prepared with each preparatory batch from the primary or an independent source, which combines a portion, or all of the elements being analyzed for calculation of precision and accuracy to verify that analysis is being performed in control.

Laboratory Duplicate Samples: identical splits of individual samples that are taken and analyzed by the laboratory to assess method reproducibility.

Laboratory Fortified Blank (LFB): a term used in conjunction with EPA/600/4-88/039 method 524.2, which describes an aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is similar to an aqueous LCS.

(m/z): mass-to-charge ratio. Synonymous with (m/e).

Matrix: the material of which the sample to be analyzed is composed. Typically, refers to water, soil/ sediment, or other environmental medium. "Matrix" is NOT synonymous with "phase" (liquid or solid).

Matrix Duplicate/Laboratory Duplicate (DUP): two representative aliquots of the same sample matrix subjected to identical analytical procedures in order to assess the procedural precision of the method through the calculation of relative percent difference (%RPD).

Matrix Spike (MS): also referred to as a Laboratory Fortified Sample Matrix (LFM). An aliquot of sample matrix (soil or water) fortified with known quantifies of specific compounds and subjected to the entire analytical procedure in order to assess the appropriateness of the method to the matrix through calculation of the percent recovery, or other accuracy term.

Matrix Spike Duplicate (MSD): a second aliquot of the same matrix as the matrix spike that is fortified also in order to determine the precision of the method.

Medium/Media: refers to the basic material composing an environmental sample or an environment of regulatory concern, i.e., water, soil, or air. "Medium" is singular; "media" is plural. This term derives from the conventional definition: "the element (earth, water, air, or fire) that is the natural habitat of an organism)."

Method: a body of procedures and techniques for performing an activity systematically presented in the order in which they are to be executed.

Method Blank (MB): also known as Reagent Blank (RB), or Laboratory Reagent Blank (LRB). A volume of ASTM D 1193 Type II (polished) water prepared in the same manner as samples. This sample is used to evaluate if cross- contamination or any memory effects are present.

Method Detection Limit (MDL): minimum concentration of a substance that can be measured and reported.

Method of Standard Additions (MSA): the method of standard addition may be required to compensate for matrix effects. This technique should not be used for interferences that cause baseline shift. The standard-addition technique involves the analysis of the unknown sample and unknown plus known amounts of standard with extrapolation of this internal calibration curve to the baseline.

- **g/kg:** a unit describing the concentration of substances within the mass of a solid medium (weight) (ppb = parts per billion).

- **g/L:** a unit describing the concentration of substances within the volume of a liquid medium (ppb = parts per billion).

mg (milligram): unit of measure for mass (weight) (1,000 mg = gram).

mg/kg: a unit describing the concentration of substances within the mass of a solid medium (weight) (ppm = parts per million).

mg/L: a unit describing the concentration of substances within the volume of a liquid medium (ppm = parts per million).

mg/m^3: a unit describing the concentrations of dusts, gases, and mists in a measured amount of air.

Mixed Waste: waste material containing hazardous chemical and radioactive constituents.

Multimedia: containing or involving more than one medium.

National Oil and Hazardous Substances Contingency Plan (NCP): this is the rule that implements the regulatory requirements of CERCLA and SARA. It guides the determination of the sites to be corrected

under the Superfund program and the program to prevent or control spills into surface waters or other portions of the environment.

National Priority List (NPL): the USEPA's list of the most serious uncontrolled or abandoned hazardous waste sites identified for possible long-term remedial action under Superfund. The list is based primarily on the score a site receives from the Hazard Ranking System. USEPA is required to update the NPL at least once a year.

Negative Pressure: indirect pressure applied to the liquid (or gas) in the form of a vacuum drawing the liquid through a filter membrane.

Onsite/Offsite:

- Onsite--within the site boundaries.
- Offsite--outside the site boundaries.

Organic: (1) referring to or derived from living organisms. (2) In chemistry, any compound containing organic carbon.

pH: a numerical designation of relative acidity or basicity (alkalinity). A pH of 7 indicates neutrality; lower values indicate increasing acidity; higher values indicate increasing alkalinity.

Physical Soil Analysis: an analysis used to determine the physical and engineering properties of a soil. Possible analyses may include particle size, dry weight, Atterberg limits, pH, redox potential, mineral class, organic carbon and clay content, density, soil porosity, compaction, and consolidation.

Positive Pressure: pressure that is applied directly on a liquid, forcing it through the filter membrane.

Practical Quantitation Limit (PQL): minimum concentration of a substance that can be reported based upon the analysis of a project-specific matrix.

Precision: agreement among the results from a set of duplicate analysis, regardless of the true value.

Preservation: methods used to retard degradation of chemical analytes within samples by inhibiting decomposition by biological action and chemical reactions, and reducing sorption effects. Methods include limiting headspace; chemical, acid, or base addition; protection from light, cooling, etc.

Professional Judgement: the ability of a single person or team to draw conclusions, give opinions, and make interpretations based on measurement results, knowledge, experience, literature, and other sources of information.

Purge-and-Trap Device: analytical technique used to isolate volatile (purgeable) organics by stripping the compounds from water or soil slurry by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto a gas chromatographic column.

Purging: removing stagnant water from a well that may bias the representativeness of the samples. Purge volume usually varies between three and five times the volume of the well.

QA Laboratory: the USACE Division or referee laboratory responsible for the analyses of the project QA (split) samples.

QA Sample/Split: a sample that is a collocated or homogenized replicate of a field sample, except that the sample is sent to the Government QA or referee laboratory for analysis. Sample receipt allows early detection of sampling, documentation, packaging, and/or shipping errors. Data comparison to contractor's data allows an assessment of primary laboratory's performance.

Quality: the totality of features and characteristics of a product or service that bear on its ability to meet the stated or implied needs and expectations of the user.

Quality Assurance (QA): an integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement that measures the degree of excellence of environmental data and communicates the information to a data generator or data user in a convincing manner.

Quality Assurance Project Plan (QAPP): the portion of the SAP that defines the laboratory analytical and chemical data reporting requirements.

Quality Assurance/Quality Control (QA/QC): a system of procedures, checks, audits, and corrective actions to ensure that all research, design, performance, environmental monitoring and sampling, and other technical and reporting activities are of the highest achievable quality (EPA).

QC Reference Standard: refer to LCS.

QC Sample: a field replicate (duplicate) sent blindly to the Contractor's (primary) laboratory. Results assess the sampling precision and handling techniques.

Quality Control (QC): the overall system of technical activities that monitor the degree of excellence of environmental data so that stated standards or requirements are achieved.

Quantitation Limit (QL): the minimum concentration of an analyte in a specific matrix that can be identified and quantified within specified limits of accuracy or precision.

Redox: oxidation-reduction potential.

Relative Percent Difference (RPD): calculation used to compare two values and assess against method precision criteria. Refer to Appendix I for further information.

(Relative) Response Factor (RF/RRF): a measure of the relative mass spectral response of an analyte compared to its internal standard. RF/RRF are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RF/RRF is calculated from the following equation:

$$RF = \frac{(A_x C_{IS})}{(A_{IS} C_x)}$$

where:

A_x = area of the characteristic ion for the compound being measured

C_{IS} = concentration of the specific internal standard

A_{IS} = area of the characteristic ion for the specific internal standard

C_X = concentration of the compound being measured

Release: any spilling, leaking, pumping, pouring, emitting, emptying, discharging, injecting, escaping, leaching, dumping, or disposing into the environment excluding any release that results in exposure to persons solely within a workplace; emissions from the engine exhaust of a motor vehicle, rolling stock, aircraft, vessel, or pipeline pumping station engine; and release of source, byproduct, or special nuclear material from a nuclear incident (NCP).

Remedial Design (RD): the technical analysis and procedures that follow the selection of remedy for a site and result in a detailed set of plans and specifications for implementation of the remedial action (NCP).

Remedial Investigation (RI): a process undertaken to determine the nature and extent of the problem presented by the release of hazardous substances into the environment (EPA). The RI includes sampling and monitoring and gathering sufficient information to establish cleanup criteria to determine the necessity for remedial action and to support the evaluation of remedial alternatives. The RI process is usually considered to encompass obtaining resources required for the field investigation, the field investigation itself, and the RI report.

Reporting Limit: the project-specific threshold limit established for a project for which, below a numerical value, the data are reported as nondetect (U) and presented as less than (<) a numerical value.

Representativeness: a qualitative measure of the extent to which a sample(s) acquired from a medium describe the chemical characteristics of that medium.

Reproducibility: the precision, usually expressed as variance, measures the variability among the results from replicates analysis.

Residual: pertaining to a residue or remainder, as in "residual contaminations." Amount of pollutant remaining in the environment after a natural or technological process has taken place, for example, the sludge remaining after initial wastewater treatment or particulates remaining in air after the air passes through a scrubbing or other pollutant removal process.

Resolution: also known as separation, or percent resolution. The separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smallest peak being resolved, and multiplied by 100. Method criteria for peak resolution may be established based on peak tailing factors (Figure I-1), but is normally evaluated based on analyst judgement.

Resource Conservation and Recovery Act (RCRA): refers to the Solid Waste Disposal Act as amended by RCRA. This act includes regulations governing solid wastes, which include hazardous wastes as defined under RCRA. The RCRA hazardous regulations govern all aspects of hazardous waste management

including identification and listing of hazardous wastes and standards applicable to generators; transporters; and owners of treatment, storage, and disposal facilities.

RI-derived Waste: any wastes generated during remedial investigation activities that may have come in contact with contaminated media at the site. These wastes usually include drilling cuttings, well development or purging water, personnel protective clothing, disposable sampling equipment, any decontamination wastes, or plastic used to collect cuttings.

Risk Assessment: qualitative and quantitative evaluation performed to define the risk posed to human health and/or the environment by the presence of specific pollutants.

Sample: a portion of material collected for chemical analyses. Note that a sample is identified by a unique sample number and that the term and the number may apply to multiple sample containers, if a single sample is submitted for a variety of chemical analyses.

Sampling and Analysis Plan (SAP): a submittal document comprised of the FSP and QAPP; used to define all aspects of the project sampling and analytical work to be done.

Screening Data: data generated by less precise methods of analysis, less rigorous sample preparation, or less stringent QA/QC procedures. The data generated provide analyte identification and quantitation, although either may be relatively imprecise.

Sediment: solid material settled from suspension in a liquid.

Semivolatile Organics: compounds that are amenable to analysis by extraction of the sample with an organic solvent. The term semivolatile organic is used synonymously with base/neutral/acid (BNA) compounds.

Sensitivity: a general term used to describe contract method detection/quantitation/reporting limits established to meet project-specific data quality objectives, or the capability of a method or instrument to discriminate between small differences in analyte concentration.

Serial Dilution: when a new or unusual matrix is encountered, a series of tests is recommended prior to release of results to verify that no matrix effects are occurring. The method recommends serial (1:4) dilution be run on samples with concentrations at least >10X instrument detection limit, with results agreeing within ± 10 percent of the original determination.

Sludge: any heavy, slimy deposit, sediment, or mass.

Slug Test: an aquifer test conducted by causing an instantaneous change in the water level in a well. The recovery of the water level with time is measured.

Soil: a natural aggregate of mineral grains with or without organic materials that can be separated by mechanical means.

Solids: materials that tend to keep their form rather than to flow or spread out.

Split-spoon Sampler: open-ended cylindrical tool used to collect samples by driving or pushing them into the ground. Split-spoon samplers have inside diameters ranging from 3 to 6.3 cm (1-3/8 to 2-1/2 in.) and usually consist of five parts, similar to a continuous barrel sampler.

Standard Operating Procedures (SOP): a written document detailing the process for an operation, analysis, or task with thoroughly prescribed techniques and steps, and is officially approved as the acceptable method of performance.

Subsurface: below the land surface.

Subsurface Investigation: a systematic study of the physical and chemical properties of the geologic materials, groundwater, and any waste products present in the subsurface.

Subsurface Soil: soil that underlies the defined limit of surface soil. Distinction between surface soil and subsurface soil is valid only when referring to risk posed by exposure of surface biota to contamination.

Superfund: the program operated under the legislative authority of CERCLA and SARA that funds and carries out the USEPA solid waste emergency and long-term removal remedial activities. These activities include establishing the National Priority List, investigating sites for inclusion on the list, determining their priority level on the list, and conducting and/or supervising the ultimately determined cleanup and other remedial actions.

Surrogate Compounds: also referred to as System Monitoring Compounds (SMC). Brominated, fluorinated, or isotopically labeled compounds (not expected to be detected within environmental samples) which are added to EVERY blank, sample, MS, MSD, DUP, standard, etc., undergoing organic analyses in order to evaluate analytical efficiency by measuring recovery.

Suspended Metals: The concentration of metals determined in the portion of a sample that is retained on a 0.45-• m filter. (The concentration of suspended metals may also be calculated from the difference between the total metals sample results minus the dissolved metals sample results.)

SW-846: a set of USEPA reference manuals containing specific methods/procedures for physical and chemical analyses (EPA/SW-846).

System Performance Check Compounds (SPCCs): term used in conjunction with SW-846 GC/MS methods to refer to the compounds in which the RF is evaluated against method-prescribed criteria to decide the validity of an analytical system.

Temperature Blank: a container filled with water packaged along with the field samples to allow the receiving laboratory a mechanism to accurately measure the temperature of the cooler and associated samples upon receipt. The samples do not undergo any chemical analysis.

Tentatively Identified Compounds (TICs): compounds detected in environmental samples that are not method target analytes, internal standards, or surrogates. Typically 10 to 20 of the largest unidentified peaks are subjected to a mass spectral library search for tentative identification. An additional charge may be associated with this procedure.

Thin-Wall Tube Sampler: a seamless steel tube with a diameter not less than 5 cm (2 in.) and an area ratio of about 10 percent. Common tubing used has a diameter of 5 or 7.5 cm (2 or 3 in.) and varies from 0.6 to 0.9 m (2 to 3 ft) long. The lower end of the tube is crimped to form a cutting edge. The upper end is attached to a coupling head. Thin-walled tubes are used in soft or moderately stiff cohesive soils to collect relatively undisturbed unconsolidated material.

Total Metals: concentration of metals determined in an unfiltered water sample that is preserved (acidified) in the field and transported to the laboratory, and then follows a rigorous digestion.

Total Recoverable Metals: concentration of metals in an unfiltered water sample that is preserved (acidified) in the field and transported to the laboratory, which then performs the digestion with hot dilute mineral acid. This preparation method is typically utilized for drinking water, solid environmental samples, and EPTox or toxicity characteristic leaching procedure extracts.

Traceability: the ability to trace history, application, or location of an entity by means of documentation and recorded identifications.

Trip Blank (TB): samples prepared by adding clean, analyte-free water to sample containers for aqueous volatile organics analysis. Preservatives are added to the blank, and the containers are sealed. Trip blanks are transported with empty sample containers to the site, and back to the laboratory with the environmental samples. TBs remain sealed until analyzed with the collected environmental samples. TBs permit evaluation of contamination generated from sample containers or occurring during the shipping and laboratory storage process.

Upgradient Sample: refers to background samples, with regard to upstream aqueous media (e.g., surface and ground waters).

Volatile Organics: compounds amenable to analysis by the purge-and-trap technique. The term volatile organics is used synonymously with purgeable compounds.

Wide-Bore Capillary Column: A gas chromatographic column with an internal diameter that is > 0.32 mm. Columns with lesser diameters are classified as narrow-bore capillary columns.