DoD Environmental Field Sampling Handbook

Revision 1.0

April 2013

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Preface

This handbook provides basic guidance for working-level environmental field sampling personnel to ensure the integrity of monitoring activities tied to regulatory reporting requirements. The handbook was developed as a tool for Department of Defense (DoD) personnel. In cases where a contractor provides environmental sampling and field testing services, the contractor is responsible for ensuring self-compliance with all environmental and occupational safety and health laws and regulations. In certain instances, special requirements may dictate a more rigorous detail, depending on federal, state, and local regulations. Local field sampling procedures should be compatible with the contents of this handbook. However, there is no intent for guidance herein to replace standard operating procedures, which may exist at the local level.

Issues regarding the contents of this handbook should be brought to the attention of the DoD Environmental Data Quality Workgroup, Field Sampling and Testing Subgroup.

The DoD Environmental Data Quality Workgroup (EDQW) developed this handbook to provide guidance for environmental field sampling personnel and laboratories performing services for the DoD. It is based on the *Navy Environmental Compliance Sampling and Field Testing Procedures Manual* (August 2009), and is expanded to incorporate DoD-wide field sampling and testing guidance. Nothing in this document relieves any field or laboratory personnel from complying with more stringent contract specifications, host-nation final governing standards, or federal, state, and local regulations. This handbook can and should be supplemented by project-specific requirements. The DoD EDQW strongly encourages project teams to involve laboratories, project chemists, and field personnel during project-planning activities. The involvement of the laboratories, field personnel, and project chemists is critical to the development of project-specific measurement performance criteria (MPC) and to the selection of methods capable of satisfying the MPC.

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Chapter 1. Introduction

1.1. Purpose

The purpose of the *DoD Environmental Sampling Handbook* is to provide general sampling guidance and promote consistency in how environmental samples are collected and analyzed. The handbook discusses strategies and techniques that can be employed to sample and analyze various media, including but not limited to water, soils, gas, and tissues. Applications and limitations of each sampling strategy and technique are briefly described. This handbook is not intended to be all inclusive, but it does provide guidance and discussion of basic sampling techniques. Many of the techniques discussed in this handbook have been derived from approved and accepted regulatory programs. When appropriate, references to standardized methods are provided.

1.2. Scope

This handbook describes techniques that can be used for environmental sampling and analysis at Department of Defense (DoD) compliance, restoration, or other sites. Handbook users are cautioned on possible differences between material presented here and requirements contained in control documents such as permits, licenses, state, local, and other countries' program regulations. These control documents have legal precedence and may prescribe sampling practices unique to a specific program or site. When sampling to document compliance with a control document (regulation, permit, etc.), the control document takes precedence over this handbook, and this handbook should be used as a guidance document only.

1.3. Background

Accurate, defensible, environmental sampling and field-testing results are imperative for regulatory compliance monitoring. There is great opportunity for error considering the variable conditions within the environment and the great variety of sampling equipment available. There is little tolerance for error since the validity of associated laboratory test results depends on sample integrity. Sampling personnel are key to the success of environmental sampling and testing programs upon which decisions are based.

At times, sampling personnel interface directly with federal, state, and local oversight personnel while sampling. They play sensitive roles in representing their commands and activities in this manner. At all times, success requires that sampling personnel be familiar with governing directives and their roles in relation to them, that they be properly trained and qualified, and specifically, that they have a strong appreciation for:

- The environmental sampling organization
- The importance of sampling plans
- Sampling and laboratory personnel interface
- Sampling equipment usage and maintenance requirements
- Sampling and field testing procedures
- Record and log keeping requirements
- Chain-of-custody (COC) requirements
- Personnel and equipment safety precautions
- Labeling, preservation, packaging, and shipping requirements
- Training and qualification requirements.

This handbook responds to the above concerns by providing clear, concise, and consistent guidance to sampling personnel. It provides the framework to identify generic requirements pertaining to sampling, to the extent possible, given the diverse missions and sites of the DoD, and is designed to augment and improve internal management of DoD environmental sampling programs. It is not intended to create any right or benefit, substantive or procedural, enforceable at law by any party against the DoD, its officers, employees, or any person.

1.4. Requirements for Laboratory Testing

An important element of the DoD's mission is to prevent environmental pollution, protect human health and the environment, and comply with regulations established by federal, state, local, and tribal governments. To document the results of the DoD's efforts in protecting human health and the environment, and to substantiate compliance with environmental regulations, the DoD procures environmental laboratory testing services. These services consist of two significant components:

- Assessing regulatory compliance of materials, systems, and processes
- Evaluating effectiveness of environmental restoration efforts.

1.4.1. Compliance Testing

The DoD has a continual need for laboratory testing services to evaluate compliance with regulatory limits defined for environmental pollutants. The DoD requires compliance testing to conform with the following environmental laws:

RCRA	Resource Conservation and Recovery Act
CWA	Clean Water Act
TSCA	Toxic Substances Control Act
SDWA	Safe Drinking Water Act
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
CAA	Clean Air Act

Note: The Emergency Planning and Community Right-to-Know Act (EPCRA) and Executive Order 12856 may place some additional demands on the laboratories.

These laws prescribe analyses of potable and non-potable water systems, hazardous and toxic materials, and air emissions. To comply with these regulations, the DoD operates its facilities pursuant to regulatory requirements that address process discharges. Permits may be required for base support systems (dry docks, sewer, water, industrial waste treatment) and production processes (painting, degreasing, abrasive blasting, flushing). The DoD accomplishes environmental analysis through both in-house and commercial laboratories.

1.4.2. Restoration Testing

Restoration testing is conducted pursuant to requirements in the following laws:

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
SARA	Superfund Amendments and Reauthorization Act of 1986
RCRA	Resource Conservation and Recovery Act

Restoration testing is performed almost exclusively by commercial laboratories. Restoration work is accomplished by using prime contractors that sub-contract analytical work to commercial laboratories. The laboratories are nominated by the prime contractor and approved by the DoD. Laboratories performing testing in support of the DoD environmental restoration programs must be accredited in accordance with the DoD Environmental Laboratory Accreditation Program (ELAP). DoD ELAP ensures laboratories performing analyses for the environmental restoration program meet a minimum quality standard. A project-specific audit of the laboratory's ability to meet project-specific requirements may also be performed. Additionally, unannounced data package audits may be completed to ensure quality data is being submitted after the laboratory has been approved.

1.5. Handbook Overview

Chapter 1 – Summarizes the handbook and gives direction for its use.

Chapter 2 – Provides an overview of the sampling program in general. This chapter is intended to provide sampling personnel with guidance concerning a sampling program, responsibilities of all personnel in the sampling program, as well as the documentation required by the United States (U.S.) Environmental Protection Agency (EPA) for each sampling event.

To sampling personnel, Chapters 3 through 12 represent the "core" of the handbook by providing the procedures and requirements of field sampling. A single sampling run will involve the following sections; see Figure 1-1:

Chapter 3 – Common Sampling Procedures

Chapter 4 –12 – Soil, Sediment, Surface Water, Storm Water, Groundwater, Drinking Water, Waste, and Process Sampling

Chapter 13 – Field Testing

Chapter 14 – Guidelines for Requesting Laboratory Testing

Appendix A – Sampler/Sampling Recommendations and Strategies

Appendix B – Requirements for Sample Containers, Preservation, and Holding Times

Appendix C – Requirements for Collection of Quality Control Samples

Of major importance with any material, is the ability to quickly locate certain information such as chapters, key words, subjects, figures, tables, etc. This handbook accomplishes this task with the following guides:

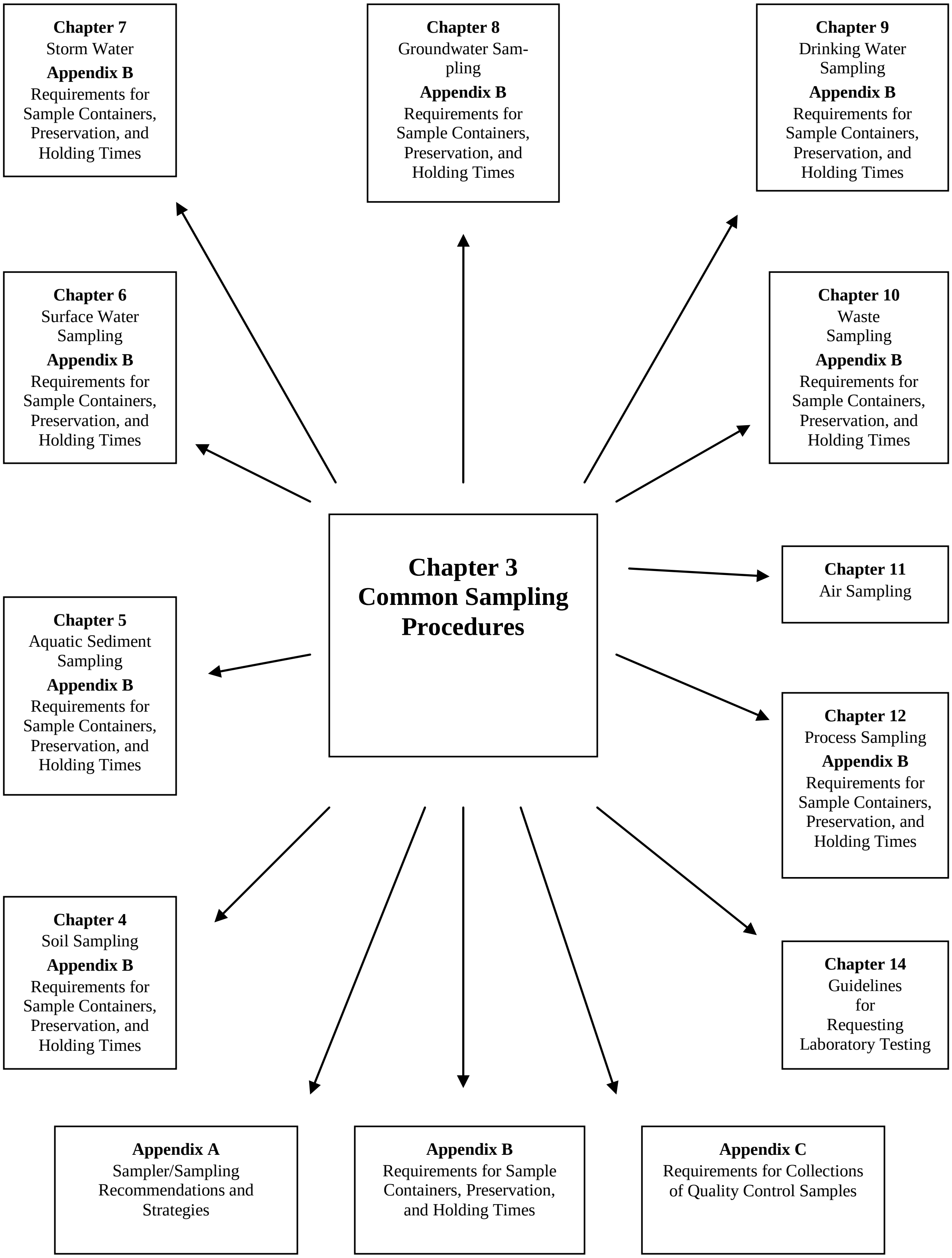
Table of Contents provides page numbers for all major sections and subsections.

List of Figures and **List of Tables** provide page numbers for all figures and tables.

Glossary of Terms and **Acronyms** define technical words and abbreviations.

Reference Sections provide sources of information used throughout the handook, including additional sources of information.

Figure 1-1. Chapter / Appendix Relationships



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Chapter 2. Sampling and Field Testing Program Overview

2.1. Purpose

This chapter presents the fundamental components of planning a sampling and analysis program and discusses methods for selecting a sampling design and strategy for collecting environmental data. In addition, guidance is provided on developing a Sampling and Analysis Plan (SAP). A well-developed SAP plays a critical role in ensuring the collection of data that have sufficient quality from which to make defensible decisions.

Planning is a necessary component of any environmental data collection program. The SAP should clearly state the purpose of the project, identify the type, quality, and quantity of data needed to accomplish project goals, and openly acknowledge any underlying assumptions or sources of uncertainty that could significantly impact the quality of the data.

2.2. Need for Proper Planning

Environmental sampling projects require careful planning and consideration by the project team to ensure that resources are effectively used to collect acceptable data for accomplishing project goals. The level of detail and amount of time spent on planning should reflect the importance and intended use of the data and the resources available to the project (sometimes referred to as the “graded approach”). In any environmental program, the three following questions need to be answered before a project is started:

- What are the objectives of the project?
- How will the data generated ultimately be used?
- What type, quality, and quantity of data are needed to accomplish the objectives of the project?

The answers to these questions provide the foundation for determining the proper sampling methodology for the project.

2.3. Systematic Planning and the Conceptual Site Model (CSM)

The Systematic Planning Process is a methodical approach recommended by the U.S. EPA to:

- Identify the technical goals of a project and site conditions
- State the expected outcome of data collection
- Outline the cost and schedule of the project
- State the acceptance criteria for the final result.

The essence of systematic planning is accurately stating the objectives related to an environmental issue and then strategizing how best to address those objectives. Successful systematic planning identifies the key decision makers and allows all participants in the environmental project to be aware of the reasoning behind their decisions. Communication between organizations and individuals involved in an environmental project is encouraged early in the planning process. This will ensure that all participants understand the needs and expectations of the data collection effort and the questions the project is trying to answer. Early communication also provides an efficient use of program resources by requiring a thorough discussion among the entire project team of the type, quality, and quantity of data required to address project goals prior to the start of the data collection process.

A key tool used in the systematic planning process is a CSM. A CSM can be defined as a written and/or pictorial representation of an environmental system and the biological, physical and chemical processes that determine the fate and transport of contaminants through environmental media to environmental receptors and their most likely exposure models. A CSM promotes project transparency among the project team by summarizing all the available information about a site and identifying what additional information must be known to achieve project objectives. The format of a project’s CSM may vary.

A good CSM addresses the following elements as applicable to the objectives of the project:

- Nature and extent of contamination
- Geology
- Hydrogeology
- Fate and transport
- Biological and geochemical conditions
- Potential transport pathways of contamination and exposure scenarios
- Potential monitoring points
- Potential receptors
- Potential exposure scenarios and pathways
- Potential areas of unacceptable risk
- Potential target treatment zones
- Site history and intended future site use
- Other factors relevant to the understanding of contamination and related risk to human health and the environment.

During the systematic planning process, project goals are linked to the individual activities necessary to reach these goals by identifying data gaps in the CSM. The CSM then serves as the foundation for evaluating all proposed and implemented project activities.

The CSM is the basis for defining the Remedial Action Objectives (RAOs). It also should be used to expose data gaps and aid in development of the SAP.

The CSM is used onsite to focus fieldwork efforts on obtaining the information needed to remove important unknowns from the model and developing an exit strategy. As site work progresses and data gaps are filled, the CSM is updated to reflect the new information. This process allows the CSM to evolve as the project progresses. The CSM ultimately serves as a planning tool as well as a modeling and data interpretation tool. It also provides a line of communication between field personnel and the rest of the project team, which keeps all parties focused on the data collection process and achieving project objectives.

Additional information on CSMs can be found in the USACE EM 200-1-12, *Conceptual Site Models* (http://publications.usace.army.mil/publications/eng-manuals/EM_200-1-12/EM_200-1-12.pdf), and the American Society for Testing and Materials E1689-95 (2008): *Standard Guide for Developing Conceptual Site Models for Contaminated Sites* (ASTM, 2008).

2.4. Aerial Photo and Map Analysis

As part of the project planning process, current and historical aerial photos, satellite imagery, and present-day maps can provide valuable insight into past and present suspected source areas. Historical photos may be used to identify past disposal pits, ground disturbance indicative of landfills, or stained soil locations that may indicate past spills. Current aerial photos or satellite imagery may be used to identify potential seeps for sampling by locating vegetation concentrations in an otherwise dry area. Similarly, the presence of stressed vegetation may indicate potential groundwater contamination. In coastline areas, comparing past and present aerial photos can identify areas of significant shoreline erosion or aggradation. Source areas that were located hundreds of feet inland in the 1940's may now be coastline features. Current aerial photos and satellite imagery can also be used to infer soil type and relative permeability of soils present at the ground surface. Coarse drainage patterns or broad, shallow valleys may indicate higher permeability soils. Conversely, streams with steep slopes indicate low permeability soils. Current aerial photos and satellite imagery can provide information on the degree of development and industrialization in the vicinity of source areas, and can be used to determine potential receptors and identify potential sample locations.

Guidance for aerial photo analysis may be found in *Aerial Photographs in Geologic Interpretation and Mapping*, U.S. Geological Survey Professional Paper 373.

U.S. EPA's EnviroMapper for Envirofacts website (http://www.epa.gov/emefdata/em4ef.home) maps potential waste generators in the project vicinity and provides information on the types of waste being generated.

Free imagery can be found at a number of sources. The Environmental Systems Research Institute (ESRI) offers two online imagery maps (World Imagery and Bing Maps Aerial) for inclusion into ArcGIS mapping projects at http://www.esri.com/data/free-data/index.html Military installations usually have imagery available also. Army Installation imagery can often be accessed through the geographic information system (GIS) or the Department of Public Works shops. This imagery can be georeferenced and provide the basis for site sample maps.

2.5. Fate and Transport

Understanding contaminant fate and transport is an important element of the CSM. In many cases, the sampling effort or remedial action to address contamination in soil is based on the potential to impact groundwater quality or surface water quality due to the leaching potential. The transport of contaminants via leaching and migration through groundwater can be a very complex process. In order to develop a quantitative predictive tool (e.g., a model), geological, geochemical, and hydrogeological data must be available, including an understanding of seasonal and historical fluctuations in hydrogeological conditions. Similarly, an understanding of the fate and transport behavior of site contaminants and related degradation products, particularly in groundwater, is critical to understanding the potential for contaminant migration and attenuation. This information is useful in understanding the nature and extent of any remediation or treatment processes that may be required to supplement natural attenuation. These data are used for a risk assessment and developing risk-based remedial action objectives for restoration projects and useful in determining impact for compliance efforts.

2.6. Geophysical Exploration

The use of surface geophysics can be valuable in planning a sampling program particularly for choosing sampling locations at suspected source areas. Geophysical techniques include magnetic and electromagnetic surveys, ground-penetrating radar, and seismic surveys. These methods can help identify buried objects that may represent sources or sampling hazards, can help define limits of past excavations or landfills, and can clarify subsurface hydrogeology that can affect contaminant transport.

Guidance for the application of geophysics for contaminated sites is available in United States Army Corps of Engineers (USACE) Engineer Manual 1110-1-1802, *Geophysical Exploration for Engineering and Environmental Investigations* (http://synectics.net/public/file/searchresults.aspx?dsn=pub&category=USACE&subcategory=Engineer%20Manuals&title=USACE%20Engineer%20Manuals&idMenu=37839&ddlDSN=SYSTM) and U.S. EPA documents such as *Geophysical Techniques for Sensing Buried Wastes and Waste Migration* EPA-600/7-84/064 and *Subsurface Characterization and Monitoring Techniques, A Desk Reference Guide, Volume 1, Chapter 1, Remote Sensing and Geophysical Methods* EPA/625/R-93/003a, and ASTM Standard D6429 *Standard Guide for Selecting Surface Geophysical Methods*.

2.7. Data Quality Objectives (DQO) Process

The DQO process is a systematic planning tool recommended by the U.S. EPA to develop sampling designs for data collection activities that support decision making. The DQO process is a method used to structure the project planning processes in order to manage uncertainty. The DQO process is best applied when critical environmental decisions need to be made (e.g., final decision-making or compliance with a standard). The DQO process integrates the work of a multi-disciplinary team for planning action-oriented environmental data collection activities. It encourages thoughtful consideration of the following:

what decisions need to be made; what data type, quality, and quantity are needed to support the decisions; what portion of the environment (and/or what time frame) shall be represented by data; how data will be used to support the decision; and what level of decision certainty (and therefore data quality) is desired. The DQO process is iterative and the final outcome is a design for collecting data (e.g., the number of samples to collect, their locations, and the procedure for sampling). Additionally, acceptable limits on the probabilities of making decision errors can be determined if a statistically based sampling design is used. The full DQO process is described in the U.S. EPA's *Guidance for the Data Quality Objectives Process* (QA/G-4, EPA/240/B-06/001, http://www.epa.gov/quality/qs-docs/g4-final.pdf). The DQO process is a seven-step planning approach based on the scientific method. It involves the entire planning cycle for an environmental project and encompasses all the activities necessary for project completion. The seven steps of the DQO process are:

Step 1: State the Problem. Define the problem clearly, identify the primary decision maker and planning team members, and determine the available budget, personnel, and deadlines.

Step 2: Identify the Goal of the Study. Develop an appropriate decision statement, identify the principal study question, define alternative actions that could result from resolving the principal study question, link the principal study question to possible actions, and organize multiple decisions.

Step 3: Identify Information Inputs. Identify the type and sources of information needed to resolve the decision statement, identify information needed to establish the action level, and confirm that suitable methods exist.

Step 4: Define the Boundaries of the Study. Specify the characteristics that define the population of interest, define the spatial and temporal boundaries, define the scale of decision making, and identify any practical constraints on data collection.

Step 5: Develop the Analytic Approach. Construct the theoretical "If...then...else..." decision rule by combining the possible results from the study, the Action Level, the scale of decision making (Step 4), and the alternative actions (Step 2).

Step 6: Specify Performance or Acceptance Criteria. Determine the decision maker's tolerable limits on potential decision errors by identifying the decision errors and base-level assumptions. Specify a range of possible parameter values where the consequences of decision errors are relatively minor, and assign probability values for the occurrence of potential decision errors.

Step 7: Develop the Detailed Plan for Obtaining Data. Identify a resource-effective sampling design for data collection that will then satisfy the DQOs documented in the previous steps. Meeting or exceeding the DQOs is the goal of the selected sampling design. The DQO process is flexible and allows the planning team to incorporate new information into the sampling plan as it becomes available. The final outcomes of the DQO process are qualitative and quantitative statements that:

- Clarify the study objective
- Define the type, quality, and quantity of required data
- Determine the most appropriate conditions under which to collect the samples
- Specify how the data will be used to address the project objectives.

For more additional information on the DQO process, see EPA QA/G-4.

2.7.1. Sampling Strategy

Selecting the correct sampling strategy for the SAP requires consideration of project resources and an understanding of the data quality required for the project. When evaluating which sampling strategy is most appropriate for a given environmental program, project teams should assess the amount of time available to complete the project, the availability of technical experts for on-site review, the type of data and quality level required for decision making, and the amount of information already known about the site. Knowing these key pieces of information will assist project teams in determining the most appropriate sampling strategy for the project.

Sampling strategies may be categorized as "static" or "dynamic." Static sampling strategy represents a phased approach to environmental data collection. During static sampling, data are collected and analyzed and then typically documented in an interim report. After the report is reviewed, the direction of the next phase in the project is decided. The phased process generally continues until the project objectives are resolved. In contrast, dynamic sampling strategy streamlines field sampling activities by providing the data needed to make decisions on site without multiple iterations of project work plans and interim reports. The term dynamic is used because field activities are designed to incorporate changes as new information is obtained, thus accommodating the iterative nature of fieldwork at many environmental sampling sites. The "Triad Approach" incorporates dynamic work strategies, real-time measurements, and systematic planning. More information on the Triad Approach can be found at http://www.triadcentral.org/.

A primary advantage of a dynamic sampling strategy is that it allows an adaptive sampling approach by encouraging decision makers and regulators to take an active role in understanding site problems and resolving sampling issues quickly. A thorough planning process that clearly outlines how decisions will be made in the field is crucial to effectively executing a dynamic sampling program. Dynamic sampling plans are living documents that provide a roadmap for decisions that a field team can follow, and outline how subsequent site activities will progress as new information is acquired.

Although the benefits of using a dynamic sampling strategy are substantial, they are not applicable for all situations. Dynamic sampling activities often need more extensive planning up front than traditional staged or static field activities. Successful dynamic sampling plans should prepare not only for what is known about a site, but also for possible conditions that could affect the completion of the fieldwork. Quick turnaround analysis is typically needed to facilitate dynamic field activities. If on-site or rapid off-site analysis is not economically or technically feasible, a static sampling strategy may be more appropriate.

Dynamic field activities also need more field oversight, because the regulator should be involved in evaluating key technical decisions as they occur. In addition, dynamic sampling routinely requires the presence of one or more experienced technical staff in the field. Experienced staff plays a key role in the decision-making process, and their recommendations can greatly influence the direction field activities take. If an experienced technical team leader is not available to oversee the fieldwork, dynamic sampling will often be an ineffective method for data collection. In this case, a static sampling strategy, which allows time for review and careful consideration by technical experts not available for on-site activities, may be more appropriate.

For additional information on the dynamic sampling process refer to EPA/540/R-03/002 (http://www.epa.gov/superfund/programs/dfa/download/guidance/40r03002.pdf).

2.7.2. Selecting a Sampling Design

One of the primary objectives of systematic planning and the DQO process is to determine the data quality and quantity needed to address project objectives. Once the data requirements are identified, the next step in the process is to develop a sampling design or methodology that specifies the number, type, and location (spatial or temporal) of the samples to be selected for measurement.

There are two main categories of sampling designs: statistical and non-statistical. Statistical sampling designs apply sampling theory and involve random selection of sampling locations. When a statistical sampling design is used, statistical inferences may be made about the sampled population from the data obtained from the sampling locations. Non-statistical sampling designs involve the selection of sampling units based on expert knowledge or professional judgment. Data derived from non-statistical sample designs cannot be assigned a quantitative level of confidence and are limited by the degree of professional knowledge available to the project team.

2.7.2.1. Statistical Sampling Designs

The most commonly used statistical sampling designs in environmental data collection are:

- **Simple random sampling.** Sample locations are selected randomly. Locations are assumed to be independent and have an equal probability of being selected. Simple random sampling is most useful when the population of interest is known or assumed to be homogeneous. A main advantage of the simple random sampling design is that it provides statistically unbiased estimates of the mean, proportions, and variability.
- **Stratified random sampling.** The target population is separated into non-overlapping strata, or subpopulations that are known or thought to be more homogeneous (relative to the environmental medium or the contaminant), so that there tends to be less variation among the areas sampled in the same stratum than among sampling areas in different strata. Strata may be chosen based on spatial or temporal proximity of the units, or based on preexisting information or professional judgment about the site or process. This design is useful for estimating a parameter when the target population is heterogeneous and the area can be subdivided based on expected contamination levels.
- **Systematic grid sampling.** Samples are taken at regularly spaced intervals over space or time. An initial location or time is chosen at random, and then the remaining sampling locations are defined so that all locations are at regular intervals over an area (grid) or time (systematic). This design provides a practical and easy method for designating sample locations and ensures uniform coverage of a site, unit, or process.
- **“Hot-spot” sampling or adaptive cluster sampling.** Samples are taken using simple random sampling, and additional samples are taken at locations where measurements exceed some threshold value. Several additional rounds of sampling and analysis may be needed. Adaptive sampling is useful for estimating or searching for rare characteristics in a population and is appropriate for inexpensive, rapid measurements. It enables delineating the boundaries of hot spots, while also using all data collected with appropriate weighting to give unbiased estimates of the population mean.
- **Ranked set sampling.** Multiple sets of field locations are identified using simple random sampling. The locations are ranked independently within each set using professional judgment, or an inexpensive, fast surrogate measurement that is correlated to the final measurement of interest. One sampling unit from each set is then selected (based on the observed ranks) for subsequent measurement using a more accurate and reliable method for the contaminant of interest. Relative to simple random sampling, this design results in more representative samples and so leads to more precise estimates of the population parameters. Ranked set sampling is useful when the cost of locating and ranking locations in the field is low compared to laboratory measurements. To use a ranked set sampling design effectively, the ranking method and analytical method should be strongly correlated.
- **Incremental Sampling Methodology (ISM).** ISM is based on particulate sampling theory. It incorporates the relationship that exists among variability of the soil, particle sizes in

the soil, the distribution of the contaminant, and the size of the sample taken.

An incremental sample is similar to but different from a composite sample. A composite sample is a combination of discrete samples. An incremental sample is a representative sample for a given area to be sampled (i.e., a decision unit (DU)). An incremental sample is collected within a DU, whereas a composite may be collected without regard to a specific DU. Although the physical process of collection is similar, the information derived from each process is different.

A DU identifies the spatial and temporal characteristics that define the decision's domain. Populations are broken into one or more discrete DUs. The identification and delineation of the DU is one of the most important factors when using ISM.

Several benefits of using ISM include improving the confidence in the mean concentration in the DU, enabling fewer samples for shipping and laboratory analysis (which would result in cost savings), and providing improved site coverage.

When using ISM, establishing a DU is critical. In addition, this method can be difficult to use for volatile contaminants, and improper selection of a DU may "dilute" contamination that could result in underestimating the need for cleanup. To ensure achieving the proper results, consulting a statistician prior to using ISM is recommended.

For more information regarding ISM, see *Incremental Sampling Methodology*, Interstate Technology & Regulatory Council (ITRC), 2011.

2.7.2.2. Non-Statistical Sampling Designs

Non-statistical sampling designs are based on professional judgment, and are usually selected with the intent of obtaining biased data. For example, selecting specific locations where a known spill occurred will provide estimates of the "worst case" or those of the highest concentrations. Sampling locations are selected based on visual signs of contamination or previous knowledge about the site. Using a biased sampling design is most appropriate when dealing with a known contamination source. The disadvantage of biased sampling is that contaminated areas may be overlooked due to inadequate knowledge of the site or a lack of visual signs of contamination.

For additional information on sampling designs for environmental data collection, see EPA/240/R-02/005 QA/G-5S: http://www.epa.gov/quality1/qs-docs/g5s-final.pdf).

Visual Sample Plan (VSP) is a software tool that can be used to employ several of the sampling strategies previously discussed. VSP supports the implementation of the DQO process by displaying different sampling plans, linking them to the DQO process, and then determining the optimal sampling specifications to protect against potential decision errors.

This easy-to-use program is highly visual, very graphic, and intended for use by non-statisticians. The VSP software program may be downloaded from http://vsp.pnnl.gov/.

2.7.3. Background Samples

In accordance with U.S. EPA guidance, background (reference area) sampling is conducted to distinguish site-related contamination from naturally occurring or other non-site-related anthropogenic chemicals (EPA, 1989). Background chemicals can be either localized or widespread and should be evaluated during site investigations in order to differentiate between Defense Environmental Restoration Program cleanup responsibilities for site-related releases and background conditions and/or non-site-related anthropogenic sources. Both naturally occurring and non-site-related anthropogenic background levels of chemicals should be established and considered as early as the Preliminary Assessment/Site Inspection (PA/SI) phase of the CERCLA process or the Resource Conservation and Recovery Act

(RCRA) Facility Assessment (RFA) of the RCRA process.

Establishing scientifically defensible background levels early in the process can provide evidence of whether a release has occurred. This technical position requires a scientifically defensible characterization of background levels of chemicals in all relevant media that are potentially present at the site due to background conditions (naturally occurring and/or non-site-related anthropogenic). The approach is consistent with the U.S. EPA's *Role of Background in the CERCLA Cleanup Program* (EPA, 2002a), the EPA's *Guidance for Comparing Background and Chemical Concentrations in Soil for CERCLA Sites* (EPA, 2002b), and the U.S. Navy's *Policy on the Use of Background Chemical Levels* (Navy, 2004).

Background is represented by the ambient concentrations of chemicals that are not site-related or attributable to site releases or activities. This includes both naturally occurring, and non-site-related anthropogenic substances present in the environment. Ideally, background samples should be geochemically similar to the impacted portions of the site being evaluated. Soil characteristics such as soil horizon and type, pH, redox conditions, cation exchange capacity, density, total organic carbon (TOC), grain size, and moisture are lines of evidence that can be used as indicators of similarity. Also, background locations should be selected that represent similar conditions of ambient, anthropogenic contaminants. Sources of anthropogenic background chemicals that may not be specifically site related include agricultural runoff, septic systems, application of pesticides, air pollution, industrial discharges, and urban pollution. Background samples are typically taken at upstream locations or areas not thought to be impacted by the primary site being investigated.

These non-impacted background or reference areas identified for sampling may be off-site or within the designated site boundaries. In order to reduce the chance of introducing potential sampling bias, background samples should be taken using the same depth and methodologies as those used for the site investigation. This includes using the same sample equipment and procedures. It is also advisable to use the same sampling personnel and laboratory to perform the analyses. It is recommended that project teams ensure that a sufficient number of study areas and background samples be collected to perform proper statistical tests for the background evaluation. (Appropriate sample sizes can be determined using software programs like VSP or ProUCL). The sampling strategy for background should be discussed and documented during the early phases of project planning.

A variety of descriptive statistics may be developed to evaluate the background data set (e.g., mean, median, range, etc.). Various statistical tests to compare the mean and/or upper bound between the site and reference areas require a sufficient number of reference samples. Geochemical association analysis can also be a valuable tool for background investigations (Navy, 2010). Elemental association relationships can augment statistical comparisons. It is advisable that the project team consult a geochemist, chemist, geologist, and/or statistician very early during project planning to ensure the appropriate chemicals are evaluated as well as the number and location of selected background samples. These should be documented in the sampling and analysis plan, which should also specify the project DQOs and procedures that will be used to collect the data.

References:

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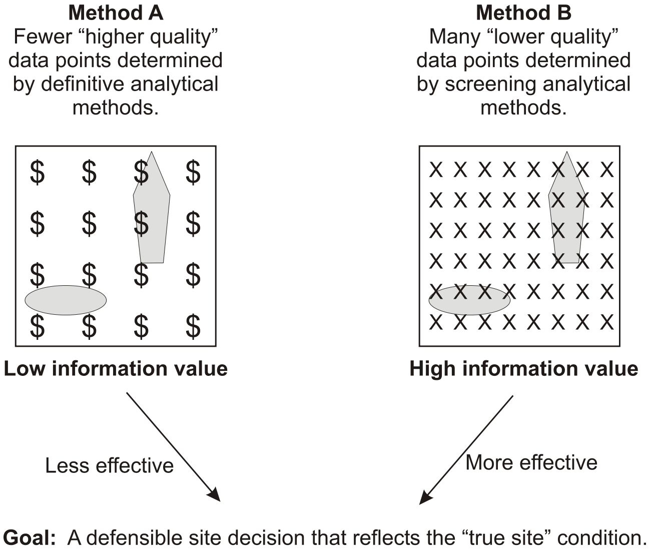
2.7.4. Collecting Effective Data

The primary objective when selecting a sampling design for an environmental program is to determine which design represents the most efficient methodology for collecting data that are scientifically valid and legally defensible. Data are acceptable when both sampling and analytical uncertainties have been managed to the degree necessary to meet accurately defined project objectives.

While the ability to document the quality of the data is significant in determining the value of a data set, the importance of the relationship between data generation and the intended use of the data cannot be overstated. For example, in a hypothetical environmental contamination problem (see Figure 2-1) a site contains two hot spot locations with significantly higher contamination concentrations than the surrounding area. The project objective is to identify where the hot spots are located. Method A analyzes a few samples using a highly accurate definitive analytical method that must be sent off site for processing. Method B surveys the area by analyzing several samples using a lower quality analytical screening method that can be performed on site. The additional data points generated using Method B represents a more effective data set than the data generated using Method A despite the fact that Method B data is determined using a lower quality analytical method. Inexpensive or faster screening methods that meet project DQOs can be a cost-effective strategy for achieving project objectives.

Figure 2-1. Data Quality vs. Information Value



Effective data reinforce a more productive conceptual framework for data generation by encouraging critical thinking about the anticipated role of data. Focusing on achieving effective data forces the systematic planning process to answer the following questions:

- What is the intended use of the data?
- What are the strengths and limitations of the proposed methods?
- What are the site-specific considerations that could adversely impact analytical performance?
- What are the site-specific considerations that will influence representative sampling?
- What are the site-specific considerations that will govern what statistical measure should be determined?

2.8. Selecting the Project Team

Systematic planning is a multidisciplinary process that requires the participation of the entire project team early in the planning stages and at key review points to ensure project resources are used efficiently. Team members should include the primary decision makers on the project as well as the end users of the data to encourage clear communication of data requirements and limitations for achieving project goals. Systematic planning evolves as new information becomes available. The involvement of the appropriate project team members at each stage in the process is necessary to keep the focus on collecting the most effective data for accomplishing project objectives. Ultimately the planning process is a collaborative effort that involves the regulator, program managers, field sampling personnel, laboratory personnel, facility health and safety officers, and any additional individuals involved in the data collection process. Encouraging active participation of the entire project team is the key to successful environmental sampling programs.

2.9. Developing a SAP

A SAP is an integral part of the systematic planning process. In general, a site-specific SAP shall be completed, reviewed, and approved prior to any data collection. In many cases, the SAP is not completed before sampling begins and results in the production of data that may not be usable for their intended purpose, thus wasting valuable project resources. It is imperative that project teams invest the time to complete the DQO process and prepare a SAP prior to data collection to increase the likelihood that "effective data" will be generated for the project.

At a minimum, each SAP should include the following sections:

- **Introduction.** Explains the objectives of the sampling program at the site
- **Objective.** Presents the project objectives, including the decision criteria applicable to each parameter analyzed, and describes how the work will meet these objectives. Explains the sampling operation in terms of the number of personnel, education, training, technical knowledge, and experience needed to meet assigned functions
- **Site Description.** Provides a description of the site, including location, current and historical uses, potential sources of contamination, and site maps showing the existing features
- **Conceptual Site Model**. Provides information on both the geology and hydrology at the site, information on existing water quality and pollutant information, and any known or potential data gaps
- **Sample Collection**. Explains the types of samples to be collected, intended methods for collection, sample handling procedures (including bottle types, preservation, filtration, and chain of custody (COC)), analytical methods, and analytical laboratories
- **QA/QC.** Includes DQOs, field QA/QC procedures (including sampling equipment construction, duplicate samples, field blanks, trip blanks), and requirements for precision, accuracy, and completeness
- **Documentation of Field Activities**. Includes field observations and procedures, sample labeling, and photographic documentation (if available)
- **References.** Individuals responsible for preparing the SAP should conduct a site walk over to familiarize themselves with the field conditions and, if necessary, consult with experienced field personnel to obtain input on appropriate sampling procedures that may be unique to the site. In addition, the SAP should incorporate any local regulatory agency requirements for surface water sampling and sampling waste management.

SAPs should be prepared based on the regulatory requirements, the information required for making decisions, and qualifying factors of the sampling and laboratory operations. SAPs describe the quality system requirements appropriate to meet the type, range, and scope of the sampling and testing being performed. Defense Environmental Restoration Program (DERP) projects must create a SAP following the UFP-QAPP. The UFP-QAPP format contains all technical and quality aspects for the life cycle of the project, including planning, implementation, and assessment. Although only mandatory for DERP projects, the UFP-QAPP format can also be used for other environmental sampling projects. Additional information and guidance on the UFP-QAPP can be found at http://www.epa.gov/fedfac/documents/qualityassurance.htm.

2.10. Health and Safety Plan

The Health and Safety Plan (HASP) is a separate deliverable sometimes included with the SAP. A site-specific HASP meeting the requirements of 29 CFR 1910.120(a)(1)(ii) or (iii) will be included when the scope of work requires it. A much less extensive site-specific safety plan will be included for the usual compliance type sampling that does not fall under 29 CFR 1910.120.

2.10.1. Service-Specific Safety and Occupational (SOH) Health Program

Navy: OPNAVINST 5100.23G, *Navy Safety and Occupational Program Manual*, is the primary reference document describing the Navy SOH Program. All elements of this handbook apply to protecting the health and safety of Navy DoD environmental compliance personnel. The relationship between OSHA standards and the Navy SOH Program is detailed in Chapter 16 of OPNAVINST 5100.23G.

Army: Army Regulation (AR) 385-10, *The Army Safety Program*, implements requirements of the OSH Act of 1970 as implemented in EO 12196, 29 CFR 1960, DODI 6055.1, DODI 6055.4, and DODI 6055.7. It provides the policy on Army safety management procedures with special emphasis on responsibilities and organizational concepts. This AR applies to the Active Army, the Army National Guard/Army National Guard of the U.S., and the U.S. Army Reserve, unless otherwise stated. It also applies to Army civilian employees and the U.S. Army Corps of Engineers and Civil Works activities and tenants and volunteers in accordance with Section 1588, Title 10, U.S. Code and AR 608-1.

Army Corps of Engineers: EM 385-1-1, *Safety and Health Requirements Manual*, prescribes the safety and health requirements for all Corps of Engineers activities and operations and applies to occupational exposure for missions under the command of the Chief of Engineers, whether accomplished by military, civilian, or contractor personnel.

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Chapter 3. Common Sampling Procedures

3.1. Purpose

This chapter presents specific sampling procedures and items common to the sampling events covered in Chapters 4 – 12. Section 1.5 and Figure 1-1 of this handbook illustrate the relationships among these chapters. Additional references include facility standard operating procedures (SOPs) and special requirements contained in regulatory programs and site permits.

3.2. Preparations for Field Sampling

The success of a field sampling program depends on the level of preparation prior to entering the field. Implementation of the SAP begins with preparing for the field sampling operation. The following preliminary steps are vital to the success of the project:

- **Preliminary Off-Site Evaluation.** Prior to implementing the SAP, the Program Manager and Health and Safety Supervisor should review any Historical Overview and Site Description sections of the SAP. This review may result in the decision for an on-site evaluation to assess the sampling procedures, relevant safety equipment, and PPE.
- **Equipment Verification.** The SAP should specify an equipment list, including sampling equipment, sample containers, and PPE. This list should be reviewed in detail by the entire sampling team and the Health and Safety Supervisor to verify that necessary items are included and appropriate for the site being sampled.
- **Inventory.** The Equipment Technician (however named) shall gather all the specified equipment and containers into one place and verify that it is on hand. Reagents, supplies, and quality control materials shall be checked and verified as appropriate. The designated technician shall notify the Program Manager that equipment preparations are complete.
- **Sign-Over of Materials.** The designated individual shall check the equipment inventory, and sign for custody if required.
- **Staffing and Scheduling.** The Program Manager shall consider the impact of specified sampling requirements on staff and schedule.
- **Screening or Field Measurements**. Sample screening or field testing for pH, dissolved oxygen (DO), sulfite, conductivity, disinfection chemicals, and temperature require additional field time. The need for additional personnel is based on time demands, training requirements and degree of difficulty. Significant field testing requirements may justify the procurement of a field laboratory and a trained field chemist to relieve other team members of this responsibility.
- **Preservation.** Preservation, either chemical or thermal, is required for most water samples. Thermal preservation usually requires icing the samples after collection and storing samples at ≤ 6° Celsius (C). For chemical preservation, two practices exist for adding preservative: 1) addition of the chemicals to the samples in the field, and 2) addition of the chemicals to the sampling containers prior to sending the containers to the field. Adding the reagents to the sample containers at the time the samples are collected requires the sampler to maintain records of addition and quality of the reagents and to follow proper chemical handling techniques. In some cases it may be advisable to have the laboratory add the reagents to specially labeled sample containers before they are sent to the field. This may reduce the fieldwork required and the possibility of field error resulting from contaminating the preservatives. Addition of the correct amount of preservative can be estimated for samples collected on a routine basis having little to no outside environmental or process effects.

WARNING: When using containers filled with preservative, use caution when filling the bottles to ensure the preservative is not released to the environment and the correct amount of preservative has been added to adequately fix the sample.

- **Time.** Many samples have short holding times prior to analysis. Review the holding time requirements and coordinate the schedule with the laboratory so the samples are analyzed within the required holding times. Holding times are dictated by the regulatory program and data may be invalidated if holding times are not met.

Note: Refer to Appendix B of this handbook for specific information on hold times, preservation, and containers.

3.2.1. Preparing for a Sampling Event

Preparing for a sampling event requires planning and a thorough knowledge of the regulatory program. The key elements for such preparation include:

- **Objectives.** The objectives should be thoroughly understood by all sampling personnel prior to sample collection. Knowledge of the compliance scope, boundaries, geography, and area roads and bridges will facilitate sampling.
- **Map of Study Area.** A map of the study area is essential for sampling. The map should be detailed enough so that sample locations and landmarks are clearly identifiable.
- **Permits and Regulations.** The person collecting samples should have a working knowledge of applicable permits, required monitoring, and other specified conditions. Regulations that potentially impact the sampling area, such as right of entry, should be reviewed by the sample collector.
- **Waste Sources.** When the objective of a project is to determine the nature, extent, or impact of a waste source upon an environmental medium, knowledge of waste source(s) within the area, as well as those sources upstream or upgradient that may impact the area, is essential. This knowledge entails knowing waste source discharge points or areas, type of waste, volume of discharge, and constituent concentration. When this information is not readily available, it may be necessary to collect background information.
- **Environmental Medium Characteristics.** If the study is of a waterway, the physical characteristics of the waterway should be known prior to sample collection. These important physical characteristics include whether the receiving waterway resembles a lake, reservoir, pond, small stream, or a river. Average and maximum recorded flow, width and depth, type of benthic substrate, and type of predominant aquatic vegetation also should be noted.
- If the study area is limited to land, it is important to have knowledge of the terrain, soils classification, geology, terrestrial vegetation, industrial and residential development, predominant land use, and wildlife.
- **Sampling Information.** A sampler must know the types of samples to be collected, (e.g., water, wastewater, soil, or solid waste). The sampler also must know whether the samples are to be collected nocturnally or during the daytime, and where within the environmental medium the samples are to be collected (including both horizontal and vertical collections) as well as the preferred method of collection.
- **Laboratory Arrangements.** Arrangements must be made with the analytical laboratory to ensure that the laboratory is expecting the samples when they arrive and has a description of the types of samples (e.g., liquid, semi-solid, solid, or biological), an approximation of the number of samples for each sample type, and the analyses requested on each sample type. Arrangements must be made for the appropriate number of sample containers and preservatives where required. Regulations on transportation of samples from the point of collection to the laboratory must be considered, and the COC record must be traceable, as detailed in the SAP.

- **Equipment.** Prior to going to the sampling location, the sampling gear should be examined to ensure that it is appropriate for the task and in good working order. Verify that any preventative maintenance has been completed according to the SOPs. Label, mark, and otherwise identify all equipment, instruments, reference materials, and associated supplies for measurement processes to indicate calibration or standardization status. Expiration dates of reagents and solutions should be checked and verified as to usability. If a boat is required, an appropriate boat, motor, and life jackets must be available, and preliminary boat launch locations should be known before going to the sampling site. All equipment should be examined prior to starting the sampling event.

Note: When in use, sampling equipment should be anchored to prevent loss in the event the rope or equipment slips through the hands of the sample collector.

- **Safety.** The safety of sampling personnel is paramount. During wading operations, a rope should be attached to the sampler and extended to an anchored person on shore. In boating operations, at least two people should be present, one to collect the samples and another to operate the boat motor. Boat personnel are required to wear life preservers and take care to avoid overloading the craft. When collecting samples, beware of snakes, stinging insects, ticks, or other animals that may cause injury to the sample collectors.
- **Personnel Transportation and Lodging.** The Program Manager must consider arrangements for transporting sampling personnel and equipment to the sampling site, and for lodging when the sampling extends beyond a working day.

3.2.2. Preliminary On-Site Evaluation

When sampling for the first time at a new location, a preliminary on-site evaluation should be conducted prior to the sampling event to ensure that all aspects of the sampling process are addressed.

Upon arrival at the site, the Program Manager (or designee) and the Health and Safety Supervisor shall check with facility personnel to determine whether there have been any recent changes at the sampling locations that would influence the SAP or modify the expected hazards.

3.2.3. Preliminary Site Safety Evaluation

After a preliminary hazard analysis, sampling locations should be inspected to develop the Safety Plan or HASP as appropriate to the scope of the project. PPE information specified may not be completely reliable, and additional air monitoring may be required. When air monitoring activities are needed, focus first on identifying conditions that present an acute health hazard, and then evaluate exposure to chemicals such as carcinogens that could create long-term health problems.

If samples are to be collected in a confined spot, testing the air within the space for oxygen content should be a priority. Tests for explosive levels of flammable vapors should be conducted next, followed by testing for the presence of hazardous concentrations of specific toxic agents (depending upon the nature of the space and its contents or previous contents).

Note: Real-time instrumentation is available for making these measurements. Air samples should be collected to evaluate the levels of other chemicals in the air that may require respiratory protection. Some organic chemicals such as gasoline vapors can be monitored with standard field instruments. However, monitoring for carcinogens will normally require the use of a field gas chromatograph or the collection of test samples for laboratory analysis.

In general, the air monitoring program to evaluate worker exposures to toxic chemicals should be designed by an industrial hygienist familiar with the facility and potential hazards to which the field sampling team will be exposed.

Review physical hazards that may be present at the site, such as unstable footing near river embankments, water safety practices, first aid supplies, equipment safety practices, and other physical hazards.

3.2.4. Explosive Safety Evaluation

The possibility of encountering explosive hazards must be considered in all sampling plans. When the presence of energetic materials is suspected from the history of a site, appropriate precautions can be incorporated during the planning stages.

Consideration should also be given to situations that can lead to the formation of unstable materials from constituents that are not originally energetic compounds. Formation of peroxides in ethers and metal picrates are two examples that have been known to create safety hazards.

3.2.5. Preliminary Sampling Evaluation

Sampling locations should be inspected to ensure the information in the SAP is correct. All equipment should be checked prior to mobilization and the day before the sampling event to ensure proper equipment operation, parts, and records are available for the sampling operation. If needed, preventative maintenance should be performed.

Reagents, supplies, reference materials, and consumable materials should be verified as to expiration dates, quality, and applicability to the assigned equipment.

Locate all the sample locations during the on-site evaluation to determine site accessibility with the designated equipment, sample location, and possible background contamination for the contaminants of interest. Electromagnetic interferences, volatile air pollutants from locations off site, weather, and climate may affect the sampling event and should be planned for, as much as practical, to avoid delays in sampling.

3.3. The Sampling Event

A typical sampling event should include the following sequential activities:

- Complete all preparation and preliminary evaluation activities as needed
- Arrive at the sampling site with appropriate equipment, supplies, materials, and sample containers
- Set up equipment, work areas, and safety areas, as described in the SAP
- Collect samples at the locations specified in the SAP or reference procedure
- Immediately following sample collection, ensure that each sample container is labeled as described in the SAP. The sample label must be traceable to the sample number, date/time sampled, sampler's name, preservative, and site name, location or unique project identifier.
- Document the exact location of the collected sample(s) in the field logbook or field notebook (FLB/FN). Also, record in the FLB/FN other observations of environmental conditions that could affect or contribute to knowledge of the sampling area and the environment where the sample is collected. Prevailing weather conditions at the time of sampling should also be recorded.
- Preserve or ice samples as appropriate and record preservation method
- Perform field tests or field screening measurements and record all observations in the FLB/FN
- Complete the COC record and other field records
- Pack and seal the shipping container with collected samples, and transport the shipping container with the COC record and any laboratory required forms to the laboratory. Retain copies of all transmitted forms.
- Return all forms and copies of relevant FLB/FN pages to the Program Manager or designee
- Clean sampling equipment for the next sampling event or storage
- Breakdown all work area and safety areas as required and return the site to the condition found at the start of the sampling event
- Dispose of all waste materials using appropriate procedures.

3.4. Sampling Procedures

The SAP refers to detailed sampling procedures or includes the details of the sampling operation. A standard SOP format should be used to incorporate the following items for each type of sampling operation:

- Sampling locations, sample numbers or identifiers
- Type, volume, and number of sample containers to be filled at each sampling location and the records to be maintained
- Contaminants to be measured and special handling procedures to ensure proper collection
- Safety, health, and hazard cautions
- Sampling equipment (construction material, type, etc.) and records to be maintained for status, maintenance, and corrective action
- Step-by-step sample collection procedures (grab, composite, continuous for specified period, etc.)
- Sampling frequency for repeated sampling at the same sample location
- Special sampling requirements (e.g., the collection of initial runoff samples after a rain for contamination)
- Sample handling procedures for each sample container (e.g., screened, filtered, sequence for filling groundwater sample containers, etc.)
- Preservatives required for each sample container and contaminant
- Reagents, supplies and support services quality, verification and validation criteria to ensure materials are used properly
- Equipment decontamination procedures to be used between sample locations and between sampling events
- Recordkeeping requirements, documentation handling, and retention requirements
- Sample, equipment, and materials storage requirements
- Provisions for storage or disposal of wastes generated during field sampling.

3.4.1. Sampling Strategies

See Appendix A for sampler and sampling recommendations and strategies for waste materials. Sampling strategies for drinking water, wastewater, groundwater and TSCA materials are permit or compliance dependent. The Scope or Purpose section of the sampling procedures should describe the rationale for the sampling strategy to ensure that all personnel involved with the project have an understanding of the sampling event.

3.4.2. Sampling Procedure Checklist

Following is a checklist of the minimum steps to address in SOP format.

- **Sampling Approach**
    - Objective
    - Design of sampling plan
    - Statistics
- **Material to be Sampled**
    - Physical state
    - Volume
    - Hazardous properties
    - Composition
- **Site**
    - Accessibility
    - Waste generation and handling
    - Transitory events, startup, shutdown
    - Maintenance
    - Climate
    - Hazards
- **Equipment**
    - Maintenance
    - Preparation and cleaning
    - Operation
    - Calibration and standardization
- **Sample Handling, Transportation, Storage and Preservation**
    - COC
    - Seals
    - Forms
    - Containers
    - Preservatives, reagents, and supplies
- **QA/QC**
    - Controls on process
    - Audits
    - Training
    - Samples, blanks, duplicates, and spikes
- **Health and Safety**
    - Personnel protection

  - Safety procedures
  - Emergency procedures
- **Laboratory**
  - Document transfer
  - Sample arrival schedule, transfer
  - Sample preservation, handling and storage
  - Analytical methods and QC
  - Reporting format and schedule.

3.5. Sample Documentation and COC Procedures

Thorough documentation of a sample's custody is required to support sample validity. The documentation must verify that the samples are representative, were collected in accordance with the requirements of the SAP, and are not vulnerable to tampering before being received by the laboratory. The COC begins when the sample is taken and ends when the sample is disposed of. Sample documentation and COC procedures include the following.

- A completed sample collection label attached to all sample containers
- Records of sampling operations written in FLB/FN or related forms as designated for the operation in the SAP. Records include sample type, sample matrix, sampling method, field test methods, and QC procedures. A table may be used to present this information.
- Identification of every sample container on a COC record and all custody transfers documented
- Custody of the samples with all discrepancies in the field operations resolved or duly recorded.

The following should be used to generate the required sample documentation.

Note: EPA's "Field Operations and Records Management System II Lite (FORMS II Lite^{TM})" software is an electronic COC and may also be used to simplify and accelerate the sample documentation process.

3.5.1. Pre-Assigned Sample Numbers

Each sample consists of all of the material collected for analysis at one place, at one time, and of one matrix, except for composite samples, which may contain components collected at different locations or time.

The Program Manager shall establish a system for assigning a unique sample number to each sample collected in the field. The numbering system will be defined in the SAP, in case additional samples are generated in the field. The number for each sample will be used to identify the sample in the FLB/FN, on the sample container, and on the COC record. The number may be used on other forms and reports presenting measurements, test data, or evaluations.

The sample numbers of field QC samples like a field duplicate should be transparent to the laboratory. The sample numbers should not reveal whether a sample is a blank sample or two field samples are duplicate/split pairs to avoid potential biasing of analytical results.

The sample number provides a common identifying code for all of the analytical results for a single sample. This is particularly useful when the results are entered into a computer database, which should include:

- Sample number
- Sample container number
- COC record number
- Matrix
- Location
- Sample type
- Sample date and time
- Sampler's name
- Parameter
- Analytical result
- QC data
- Compliance limit and
- Data qualifier code (optional).

Results from analysis of trip blanks, field blanks, equipment decontamination blanks, split samples and MS/matrix spike duplicate (MSD) samples may be entered into a computer database. In some testing programs, these results are used to generate the data qualifier code for the analytical results from test and duplicate samples.

It is recommended that the information associated with each sample number consists of elements describing the sample type, matrix, location, and the time and date of sample collection as required.

Note: If the sampling and analytical data are to be added to an existing database, sample numbers should be consistent with database requirements.

3.5.2. Sample Container Labeling

Sample labels are an important part of proper documentation to reduce the possibility of confusing sample containers, and to provide the necessary handling information. Sample containers should be pre-labeled as much as practical before sample collection. The labels may be protected from the sample matrix with a clear tape covering. For volatile samples, check with the laboratory to ensure that any labels being used do not interfere with their auto-samplers. Sample labels should include sample number, date and time sampled, location, sample type, preservative and the sampler's initials or signature.

Sample numbers may be unique to the sample location, to the sample type or to the container. In some labeling processes, a unique sample number is written on the container label, and all information recorded on the accompanying form(s) is traceable to the unique sample number.

Some number schemes uniquely number each sample container. All data reported for the sample includes the sample container number for traceability to the container measured. This is useful when sample containers are cleaned and lot controlled, and traceability from container preparation, preservation, sampling, and testing is required.

A designated Field Sample Custodian or sampler should label the sample containers when they are filled. Preprinted, adhesive, multiple part labels formatted as shown in Figure 3-1 may be used. Each part includes the unique sample container number that may be pre-numbered to avoid duplication.

Note: Because 40-mL volatile organic analysis (VOA) vials may be stuck in an autosampler, the field sampling team leader needs to contact the laboratory to make sure if applying a clear tape over a sample label of 40-mL VOA vials is acceptable. Use waterproof ink to make label entries. FLB/FN notations should provide an explanation if a pencil was used to fill out the sample container label due to field weather conditions. Because waterproof ink may contain target VOAs such as xylene, toluene, or alcohols as a solvent, great care is needed to prevent VOA samples from contamination by the solvent of waterproof ink or permanent marker.

Figure 3-1. Multiple Part Container Label

PROJECT NAME

Sample #: XXXXX

Container #: XXXXX
Sample #: ________________
Date: ______ Time: _________
Location: _________________
Cont. Size: _________________
Cont. Type: _________________
Matrix: _________________
Type of Sample: ________________
Preservative: _________________
Signature: _________________

3.5.3. FLB/FN

The FLB/FN is the written record of all field data, observations, field equipment calibrations, and sample collection activities. Potential for future legal actions dictates that the FLB/FN be site-specific and that they be hardbound (e.g., ledger, composition book, diary, etc.). All pages (front and back) shall be serially numbered so removal will be apparent. Samplers shall adhere to the following guidelines when using FLB/FN.

- The FLB/FN shall be assigned to the QA/QC Coordinator or designee. Additional log books may be assigned by the Program Manager or designee to the Field Chemist and the Health and Safety Supervisor. The QA/QC Coordinator or designee shall note in each FLB the individual to whom it was assigned. The FLBs may be controlled by the QA/QC Coordinator or the Program Manager.

- Each FLB shall be annotated with the sampling program name or number.
- Key personnel and telephone numbers shall be listed on the first page.
- Entries shall be written in waterproof blue or black ballpoint pen. Avoid felt tip pens. *Do not use pencil.*
- Start a new page at the beginning of each day.
- Entries should be chronological – a time notation should introduce each entry.
- Sketch or obtain a map of the area or facility. Include sketches of layout, structural features, and points of interest or contamination. Include a north arrow and a rough scale. If possible, obtain a site map (reduced if necessary) and permanently place it in the FLB/FN.
- Language should be objective, factual, and free of personal feelings or other inappropriate terminology. Speculation or personal observations may be included if they are clearly identified as such.
- Do not erase or scratch out errors. Draw a single line through the error, then insert corrected material. The person who made the error shall initial and date the correction as well as clearly state the reason for the error.
- Entries or corrections made by individuals other than the person to whom the FLB/FN was assigned shall be signed and dated by the individual making the entry or correction. An explanation for the correction should be noted.
- The last entry for each day should include a short summary of the day's activities, weather conditions and the time the site was left. As appropriate, the last entry for each week should be a summary of the week's activities. Weekly summaries should be thorough and descriptive.
- The FLB/FN shall be signed at the end of each day. Signatures shall be written on a single diagonal line drawn across the blank portion of the page following the day's last entry.
- All FLBs/FNs shall be returned to the individual designated for review and final storage when sampling is completed as described in the SAP.
- FLB/FN entries will contain a variety of information. Information to be entered at the start of each day of sampling includes the following:
    - Date of the sampling event
    - Time sampling started and approximate time for set up of equipment
    - Weather conditions
    - Level of PPE being used
    - Names of field sampling team members and others present during the sampling.
- Fully document all deviations from the SAP or changes in sampling procedures. Problems, delays, or any unusual occurrences such as improper equipment or breakdowns should be included, along with resolutions and recommendations. Summarize the content and conclusions of all relevant meetings, discussions, and telephone conversations in which you are involved. Include the names and affiliations of all personnel involved. Thoroughly document all directives and/or guidance from EPA or other government personnel. Directives that give personnel specific authority to make critical decisions must be documented in the FLB/FN.
- Whenever a sample is collected or a measurement is made, a detailed description of the location must be recorded. The source from which the sample is collected should be clearly identified to maintain traceability and allow another person to locate the exact sampling location. The ability to relocate the sample site ensures duplication of future sampling events. Measurements from permanent features (e.g., center line of road, numbered utility pole, etc.) to the sample point must be made and entered into the FLB/FN. Coordinates on a map, or an accurate site sketch with distance measurements to known locations are other options to ensure the exact location of each sample is recorded.

- Describe the site thoroughly so another person will be able to locate the exact sample location. Note signs of contamination such as oily discharges, discolored surfaces, unusual odors, dead or distressed vegetation including types of plants, if possible. Photographs may be taken to provide evidence of visual observations, record site conditions, and assist with locating the sample site in the future. Photographs taken of sample locations should be noted along with the picture number. Log the record in the FLB/FN to identify which sampling site is depicted in the photograph.

Note: A series of photographs can be identified by taking the first photograph of an informational sign with the sampling program name, number, and the project number on it.

- Each time a sample container is filled and labeled, a copy from the multiple part form of the sample container label or reference number label with all information recorded shall be put into the FLB/FN.
- All equipment used to make measurements must be identified by type, manufacturer, and serial number, along with the date of calibration. Details of field calibration procedures and results shall also be included in the FLB/FN.
- Note decontamination or disposal procedures for all equipment, samples, protective clothing, and personnel decontamination procedures.
- For each delivery or shipment of samples to a laboratory, record the following information in the FLB/FN:
    - Custody procedures and serial numbers
    - Packing and shipping procedures (record air bill numbers)
    - Name, address, telephone number, and contact of the laboratory performing the analysis.

Note: The laboratory address should be the sample receipt address, which may not be the same as the mail receipt address.

3.5.4. Field Notes/Field Sampling Forms

Field notes or field sampling forms are used in addition to or in lieu of field log notes. When field notes are used in lieu of an FLB, the record keeping practices presented in Section 3.5.3 should be followed. The field form provides a place for the sampler to record the information required for the project. Field forms are specially designed for any given project and may be completed one per sample or one per sampling event. The forms include blank lines for recording the information necessary for the project to ensure the proper information is recorded. All blanks must be completed on a field form to ensure proper documentation. The sampler completes the field form for all samples collected including QC samples. An example of a field form for a well sampling activity is presented in Figure 3-2 below.

Note: A review of the regulatory program's specific requirements must be conducted to ensure that all documentation requirements are met. Some programs do not allow the use of loose field forms as the sole documentation vehicle and require hardbound logbooks.

The field form lists the sample number, location, matrix, the type and number of sample containers filled (including QC samples), any chemical preservatives added and checked for each sample container, sampling procedure reference, deviations to the procedures and all field measurements and observations.

The Field Sample Custodian indicates acceptance of the information on the field form by signing and dating the form. In cases where multiple part forms are used for the sample label, for each sample container filled, one part of the multiple part adhesive sample container label is placed on the field form at the appropriate location. The completed field forms are returned to the Program Manager as soon as possible and by the means indicated in the SAP. Deviations or problems encountered during the sampling event must be communicated promptly in writing to the Program Manager or designee. This may be completed by sending the field form by facsimile or other means to communicate the deviations, as well as allow for continuation of the project and ensure sample holding times are not jeopardized.

Note: The field form becomes part of the permanent project records, but is not usually sent to the laboratory.

3.5.5. Chain of Custody (COC)

An overriding consideration for environmental measurement data is the ability to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Documentation of security, field handling criteria, shipment, laboratory receipt, and laboratory custody until disposal, provides evidence of proper processing. The degree of custody documentation is dependent on the regulatory program, data use, and needs. Many state programs for sampling wastewater and drinking water do not require "legal custody," but recommend legal custody whenever data is known to be used for evidence. A review of data use and risk of legal proceedings will dictate the type of custody procedure to be employed. Documentation consists of a COC record that is completed by the Sample Custodian.

3.5.5.1. Field Custody Procedures

The Field Sample Custodian or sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples. A sample is considered to be "in custody" for legal proceedings if it is:

- In a person's actual possession
- In view after being in physical possession
- Locked up so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel only.

If any one of these is not in place at all times, the COC is broken.

The Program Manager or designee shall review all field activities to determine whether proper custody procedures were followed during the fieldwork and whether additional samples are required. The sampler or Sample Custodian shall notify the Program Manager of any breach or irregularity of COC procedures described in the SAP.

Figure 3-2. Field Form

Sheet _1__ of __1_

MICROPURGE/LOW-FLOW SAMPLING LOG

PROJECT: ____________________
EPA ID NO: ____________________
Proj./Task No. ____________________
Date: ____________________
Weather: ____________________
Samplers: ____________________
____
Purge Method: Micropurge
Sample Collection Method:
Sampling Device:
Tubing: **Pump Intake Depth:** ________
Total Recovered Purge Water This Well: ________

WELL ID: ____________________
____
Well Condition: ____________________
Well Riser Dia. (ID): ____________________
Screened Interval: ____________________
S.W.L. Measuring Pnt: ____________________
Well Bottom Depth: ____________________
____
____
STATIC WATER LEVEL: ____________________
Initial Purge Volume: ____________________
Approximate Pump Throttle Setting: ____________________

Groundwater Sample Data:

Sample ID	Analysis	Primary	QC	MS/MSD	Blank	Sample ID	Analysis	Primary	QC	MS/MSD

Instrumentation/Equipment Data:	**Calibration Date:**

Field Test Results:

Hach Kit Tests:

D.O.		mg/L
Total Iron:		mg/L
Ferr. Iron:		mg/L
CO2:		mg/L

Comments: ____________________
Observations: Clarity: Odor: Floating Product: Sheen:

PURGE WATER DATA TABLES

Stabilization Parameters	Units	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes
Temperature	C								
Sp. Cond.	ms/cm								
D.O.	ppm								
pH									
ORP (Eh)	mV								
Turbidity	NTU								
Clock Time									
Static W.L.									
Flow Rate	ml/min.								

Stabilization Parameters	Units	____ Minutes	____ Minutes	____ Minutes	____ Minutes	____ Minutes	____ Minutes	____ Minutes	____ Minutes
Temperature	C								
Sp. Cond.	ms/cm								
D.O.	ppm								
pH									
ORP (Eh)	mV								
Turbidity	NTU								
Clock Time									
Static W.L.									
Flow Rate									

3.5.5.2. COC Records

From time of collection through final sample disposal, there are many transfers of custody during the course of a sampling program. All sample containers must be accompanied by a COC record to document these transfers. A separate COC record shall be prepared by the Field Sample Custodian or sampler for each sampling event. In some programs, a COC record accompanies each shipping container and includes a pre-numbered COC record. This record lists the sample containers that are in the shipping container, and serves as the packing list for the container. The serial number on the form becomes the identifying number for the shipping package.

Figures 4-3 provides an examples of a sample COC record. The example has been used for a wide variety of regulatory programs and meets legal COC requirements. It tracks the samples from sample collection to disposal. All sampling, preservative, and test information is included. The SAP will indicate the individual responsible for completing each section. The following information relates to the numbered blocks:

COC Record–Figure 3-3.

(1) The **company/command** name and **code** for the source of the funding.

(2) The **contact name** for the Program Manager or designee indicated in the SAP.

(3) The **job order number** (**J.O. #**) is entered to trace the information to the specific job.

(4) The **signature** of the Program Manager or designee authorizing the funds.

(5) The **permit number** (**No.**), if applicable, for the samples collected. The number is issued by the regulatory agency for specific compliance reporting.

(6) The **sample ID/location** based on permit designations or actual site location name.

(7) The **sample taken date and time** are recorded for grabs on the start line only and for composites on the start date and time and stop date and time.

(8) The code for **sample type** such as grab, composite flowing and composite time (see Section 18).

(9) The initials for the **person sampled by**.

(10) The code for **sample matrix** such as liquid, solid, and gas (see Section 18).

(11) The code for **preservative** (see Section 18).

(12) The **# of samples** and **container** type are entered as “4-P” for four plastic containers (See Section 18).

(13) The **analysis** to be performed - may reference descriptions in the SAP.

(14) The field reading for **pH** for the sample containers indicated.

(15) The field reading for **temperature** with the unit of measure for the sample containers indicated. The SAP may indicate the temperature to be recorded in the outfall temperature and not the sample temperature.

(16) The field reading for **other** required measurements may be entered with the unit of measure. The SOP and name of the test must be indicated on the custody form.

(17) After the samples are preserved, the **preservation is verified**. The verification is noted per the SAP. This verification may be temperature, pH, or if all is correct an indication is made as “OK.”

(18) This section of the custody form contains **common codes** to be used by the sampler when completing the custody record. When situations arise that do not match the code designations, alternates may be added for the one time use on the custody form.

(19) The expected **turnaround** for sample request is placed in this area. The reason is presented to determine if the turnaround time is regulatory, project specific, or based on holding time requirements.

(20) **Special instructions** or comments may be entered in this space.

(21) The **regulation applied** to the project is checked.

(22) The **sample collection/charge**, **possible sample hazard** and other **comments** relate to the command in charge of sampling, special sample hazards, or to other sample comments. Reference may be made to code or specific sections of the SAP.

(23) The **delivery order number** is entered.

(24) The **contract lab** and **contract number** (**No.**) are entered for testing work performed by a designated contract laboratory.

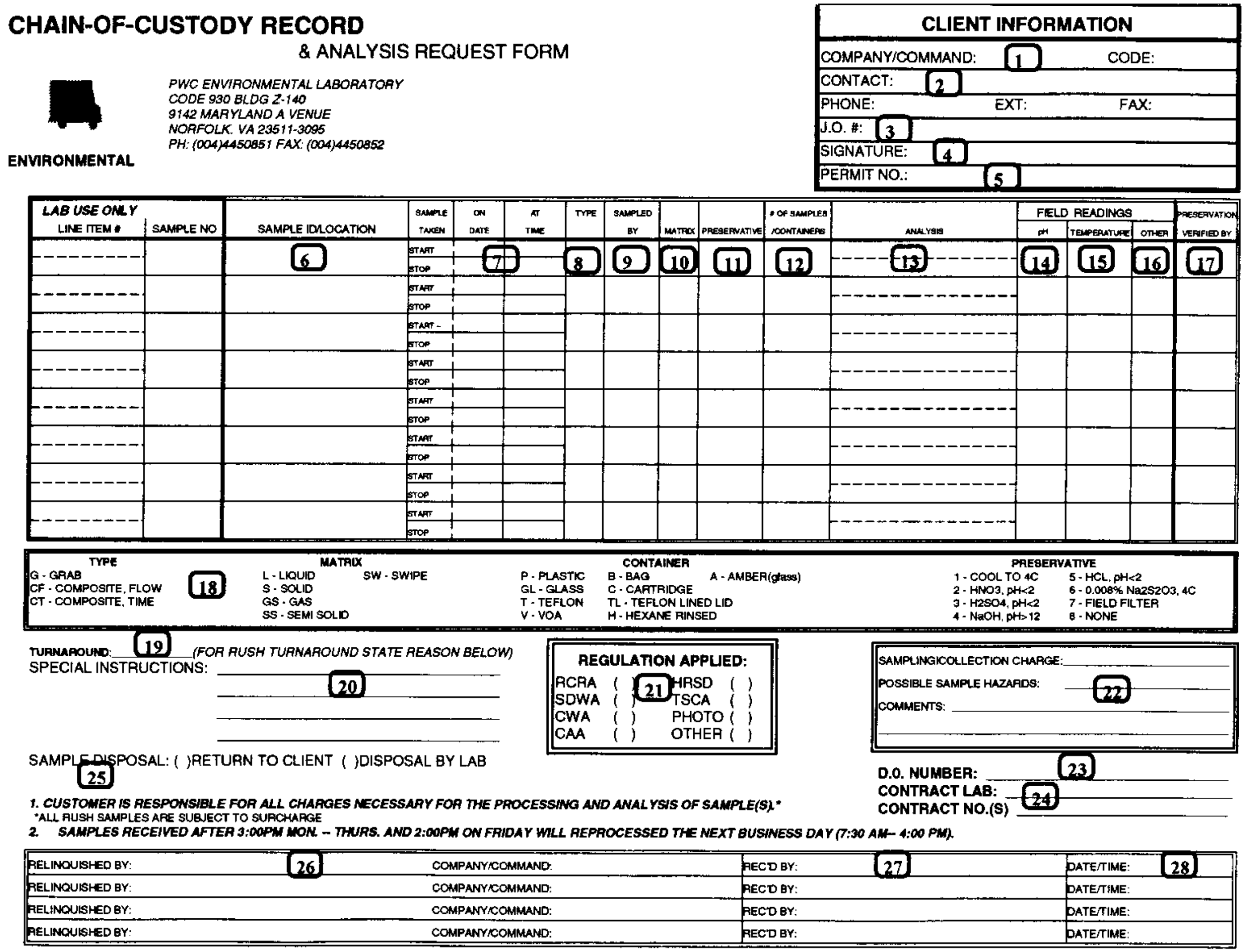
(25) The **sample disposal** method and the date completed.

(26) The signature and **company/command** of the person relinquishing custody (**relinquished by**).

(27) The signature of the person custody is **received** (**rec'd**) **by**.

(28) The **date/time** custody is transferred.

Figure 3-3. COC Record



CHAIN-OF-CUSTODY RECORD
& ANALYSIS REQUEST FORM

ENVIRONMENTAL

PWC ENVIRONMENTAL LABORATORY
CODE 930 BLDG Z-140
9142 MARYLAND A VENUE
NORFOLK. VA 23511-3095
PH: (004)4450851 FAX: (004)4450852

CLIENT INFORMATION

COMPANY/COMMAND: 1 CODE:
CONTACT: 2
PHONE: EXT: FAX:
J.O. #: 3
SIGNATURE: 4
PERMIT NO.: 5

LAB USE ONLY LINE ITEM #	SAMPLE NO	SAMPLE ID/LOCATION	SAMPLE TAKEN	ON DATE	AT TIME	TYPE	SAMPLED BY	MATRIX	PRESERVATIVE	# OF SAMPLES /CONTAINERS	ANALYSIS	FIELD READINGS pH	TEMPERATURE	OTHER	PRESERVATION VERIFIED BY
		6	START STOP	7		8	9	10	11	12	13	14	15	16	17
			START STOP												
			START - STOP												
			START STOP												
			START STOP												
			START STOP												
			START STOP												
			START STOP												

TYPE 18
G - GRAB
CF - COMPOSITE, FLOW
CT - COMPOSITE, TIME

MATRIX
L - LIQUID
S - SOLID
GS - GAS
SS - SEMI SOLID
SW - SWIPE

CONTAINER
P - PLASTIC
GL - GLASS
T - TEFLON
V - VOA
B - BAG
C - CARTRIDGE
TL - TEFLON LINED LID
H - HEXANE RINSED
A - AMBER(glass)

PRESERVATIVE
1 - COOL TO 4C
2 - HNO3, pH<2
3 - H2SO4, pH<2
4 - NaOH, pH>12
5 - HCL, pH<2
6 - 0.008% Na2S2O3, 4C
7 - FIELD FILTER
8 - NONE

TURNAROUND: 19 (FOR RUSH TURNAROUND STATE REASON BELOW)
SPECIAL INSTRUCTIONS: 20

REGULATION APPLIED: 21
RCRA ( ) HRSD ( )
SDWA ( ) TSCA ( )
CWA ( ) PHOTO ( )
CAA ( ) OTHER ( )

SAMPLING/COLLECTION CHARGE:
POSSIBLE SAMPLE HAZARDS: 22
COMMENTS:

SAMPLE DISPOSAL: ( )RETURN TO CLIENT ( )DISPOSAL BY LAB 25

D.O. NUMBER: 23
CONTRACT LAB: 24
CONTRACT NO.(S)

1. CUSTOMER IS RESPONSIBLE FOR ALL CHARGES NECESSARY FOR THE PROCESSING AND ANALYSIS OF SAMPLE(S).*
*ALL RUSH SAMPLES ARE SUBJECT TO SURCHARGE
2. SAMPLES RECEIVED AFTER 3:00PM MON. -- THURS. AND 2:00PM ON FRIDAY WILL REPROCESSED THE NEXT BUSINESS DAY (7:30 AM-- 4:00 PM).

RELINQUISHED BY: 26	COMPANY/COMMAND:	REC'D BY: 27	DATE/TIME: 28
RELINQUISHED BY:	COMPANY/COMMAND:	REC'D BY:	DATE/TIME:
RELINQUISHED BY:	COMPANY/COMMAND:	REC'D BY:	DATE/TIME:
RELINQUISHED BY:	COMPANY/COMMAND:	REC'D BY:	DATE/TIME:

The COC record identifies which pairs of sample containers were collected for the same analysis, and identifies the sample containers that were filled with sample for use as the MS/MSD QC samples. Based on the needs and data use, the COC record may not list any information as to the exact sample location or whether a sample is a field duplicate, field blank, trip blank or an equipment decontamination blank. This information is kept as blind information from the laboratory to ensure objective reporting. When this process is used, records must be maintained that trace the sample collected in the field with the sample as identified to the laboratory. Compliance data for drinking water or wastewater testing do not require blind submissions. The QC sample information is provided to the laboratory to ensure prompt notification when the QC data does not meet the SAP specifications.

Whenever samples are split with a second laboratory or government agency, a separate COC record may be prepared for the second set of samples. The additional set of COC records must be noted. Copies of the original may be sent with the split samples noted, or a separate form may be prepared by copying the appropriate information for the samples onto the additional form. In all cases, the use and need of the additional form should be noted.

Upon completion of the packing of each shipping container, the Field Sample Custodian shall confirm the completeness of the COC record by signing the COC record. If a multiple-part form is used:

- The original copy is put into the shipping container
- The first copy is sent immediately (preferably by fax) to the Program Manager or designee
- The second copy is kept with the FLB/FN or copy of the field form.

If a single part form is used, photo copies should be made for the Program Manager and the FLB.

After the COC record is completed and all samples are packaged and shipped to the appropriate locations, the person relinquishing the samples to the laboratory or agency shall request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses to sign, this is noted in the "received by" space.

Field COC terminates upon laboratory receipt of the samples. The laboratory should complete the "received by" sections and if appropriate, the "preservative checks" sections on the COC record and return the original signed record to the Program Manager. If there are any discrepancies between the COC record, the contents of the shipping container, and the SAP or contract requirements provided to the laboratory, the samples in question shall be segregated from normal sample storage, and the laboratory shall immediately notify the Program Manager. In some cases, the laboratory checks the sample submittal and recordkeeping to ensure adherence to the SAP. This added check is often used in drinking water and wastewater testing programs for compliance monitoring. Recordkeeping and information checks should be performed by the laboratory to ensure the samples received meet the requirements of the SAP.

3.5.5.3. Custody Seals (Optional)

Custody seals are narrow strips of adhesive paper used to indicate whether a shipping container has been opened during shipment. The seals are placed along the edges of the most exterior container in which samples are enclosed. It is not always necessary to place seals on individual sample containers in the shipping container.

Paper custody seals should be signed and applied before the shipping container is shipped to the laboratory. The preferred procedure includes use of a custody seal attached to the front-right and back-left of the container. Custody seals are covered with clear plastic tape. Another way to use custody seals, is to put all sample containers with packing and ice in a large garbage bag and seal the garbage bag with a signed custody seal.

3.5.5.4. Custody Transfer

Transfer of custody and shipment procedures are as follows.

- Each sample shipping container shall be accompanied by a properly completed COC record. The original of the record shall be included in the container. The Field Sample Custodian shall keep a copy of the completed form as part of permanent documentation and will send a copy of the COC record to the Program Manager.
- When transferring possession of samples, individuals relinquishing and receiving shall sign, date, and note the time of the transfer. This record documents custody transfer from the Field Sample Custodian to another person, to a mobile laboratory, to the permanent laboratory, or to a secure storage area.
- If the sample container is sent by common carrier, a bill of lading shall be used. Bill of lading receipts shall be sent to the Program Manager for permanent retention. If sent by mail, the package shall be registered with return receipt requested. Commercial carriers and the U.S Postal Service are not required to sign off on the COC record as long as it is sealed inside the package with the sample container and the custody seals remain intact (if used).

3.5.6. Request for Analysis

The Request for Analysis form is often incorporated into the COC record since the chain must accompany the samples. In more complex sampling programs, an additional form may be used to request testing.

When contracting for laboratory services and prior to submitting the samples, the laboratory should be contacted and the following information presented. The Request for Analysis form can be used as a preliminary contact mechanism to ensure that the scope of work is understood. This form:

- Specifies the analyses, procedures, and QC data to be performed on each sample container and the compliance protocols to be followed
- Specifies the laboratory accreditation/ certification required to be maintained during the period of the contract
- Authorizes the payment for the analyses
- Alerts the laboratory to any anticipated hazards associated with the samples and custody procedures to be followed while the samples are in the possession of the laboratory
- Specifies the reporting requirements and content for the final report from the laboratory
- Instructs the laboratory as to the disposition of the samples after the completion of the analyses.

3.6. Sample Packaging, Handling, and Transportation

The Field Sample Custodian is responsible for the proper field storage, security, packing, and shipping of the samples from the field to the laboratory or designated holding location. The packaging, labeling, and shipment of samples by common carrier are regulated by the DOT and the International Civil Aviation Organization (ICAO)/International Air Transport Association (IATA), when appropriate. Instructions for classification, labeling, and packaging of hazardous materials are contained in DOT regulations (49 CFR 172 and 173, and subsequent Parts). Overnight couriers generally accept materials shipped under these regulations. However, some couriers have additional restrictions for hazardous shipments. EPA also regulates the shipment of hazardous waste and hazardous material by requiring labeling on certain packages.

The procedure for determining whether a sample is hazardous under DOT regulations is complex, as is the determination of the proper shipping name, packaging requirements, and labeling requirements for DOT hazardous materials. A summary of specific requirements are addressed below. Should questions arise, assistance is available from the DOT (1-202-366-4000) and Federal Aviation Administration (FAA) (1-866-835-5322) hotlines.

Samples obtained at sites are classified for shipping purposes as either environmental (nonhazardous) samples or hazardous samples. If a material is being shipped for testing to determine its hazards, a tentative hazard class assignment should be made based on knowledge of the material. Samples requiring special packaging or labeling are those containing chemicals that are listed as hazardous materials in:

- 49 CFR 172.101
- CERCLA RQ Hazardous Substances
- DOT CLASS 9 listed in 49 CFR 172.101 Appendix A, Poison DOT Class 6.1 and Flammable Liquids.

Environmental (non-hazardous) samples are those that are not classified as Hazardous Materials under DOT regulations, are packaged in quantities less than the CERCLA RQ, *and* for which a Hazardous Waste Manifest is not required by EPA. These samples require careful packing, but no special shipping procedures. In general, samples of groundwater, surface water (other than leachate or lagoons), and soil may be shipped as environmental samples (non-hazardous) to an analytical laboratory for testing if each of the sample containers contains less than 1 pound of soil or 1 gallon of water, and the entire shipping package weighs less than 66 pounds. Eventual analysis for a hazardous constituent does not necessarily classify a sample as a DOT hazardous material, nor does the classification of a material as a hazardous waste under EPA regulations. DOT regulations forbid the shipping of non-hazardous materials as hazardous. However, if any doubt exists as to whether the sample might be classified as a hazardous material, the sample should be treated as hazardous.

Note: For details on the shipping of non-hazardous waste, refer to *ASTM D6911-03: Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis*. This standard provides guidance in determining the most appropriate procedures for packaging and shipping environmental samples.

The storage and disposal of hazardous waste is regulated by the EPA. Hazardous waste, as specified in 40 CFR 262, is not exempted from EPA manifesting requirements. However, EPA RCRA regulations exempt samples collected for analysis or treatability testing from the RCRA requirements that otherwise apply to hazardous waste (including the requirement for a Hazardous Waste Manifest). The definitions for these exemptions are:

- **Samples for Analysis**. 40 CFR 261.4(d): Samples of solid waste, water, soil, or air, which are collected for the sole purpose of testing to determine their characteristics or composition, when samples are being sent to the laboratory for testing or are being returned to the collector after testing.
- **Samples for Treatability Testing**. 40 CFR 261.4(e): Samples collected for the purpose of conducting treatability studies when they are being transported to the testing facility provided they meet criteria for the quantity of material, packaging, and permit status of the receiving facility.

3.6.1. Sample Packaging Requirements

The Field Sample Custodian is responsible for the packing and shipping of the samples from the field to the laboratory. Samples shall be properly packaged for shipment and dispatched to the laboratory for analysis with a signed custody record enclosed in the shipping container box or cooler. Shipping containers shall be locked or secured with strapping tape in at least two locations. Shipments that are sent to an on-site laboratory or one in close proximity that does not require the use of a common carrier shall be transferred in accordance with local regulations. Table 3-1 below lists sample packaging procedures that will ensure samples arrive at the laboratory with the COC record intact.

The following major issues must be addressed in preparing environmental samples for shipment to the laboratory by common carrier:

- Compliance with EPA regulations, so the samples are not classified as hazardous waste
- Compliance with transportation regulations, including use of the proper shipping containers, use of warning labels, and completion of the required paper work
- Packing, to assure that the samples do not break or leak during shipping. This includes:

  - Using approved containers meeting DOT drop test specification
  - Lining coolers or containers with plastic bags
  - For glass containers, wrapping each in bubble wrap and placing in a clear plastic resealable food bag
  - For plastic containers, placing each in a clear plastic resealable food bag
  - Never stacking glass containers or laying glass on its side.

3.6.1.1. Samples Classified as Flammable Liquid

Table 3-2 Column 1 lists packaging procedures that apply to those flammable and combustible liquids that do not meet the definitions of another hazard class except DOT Class 9, and for which exceptions under 49 CFR 173.150 are allowed. This includes Flammable Liquids Not Otherwise Specified (NOS), toluene, gasoline, and many of the other flammable liquids that are commonly encountered on hazardous waste sites.

Note: The DOT definition of "liquid" is different from that used by EPA. For purposes of transportation, liquid means a material that has a vertical flow of over 2 inches (50 mm) within a 3-minute period, or a material having 1 gram or more liquid separation, when determined in accordance with the procedures specified in *ASTM D4359-90, Standard Test Procedure for Determining whether a Material is a Liquid or Solid*, (49 CFR 171.8).

3.6.1.2. Samples Classified as Poison — DOT Class 6

Table 3-2 Column 2 lists packaging procedures that apply to those poisonous liquids and solids for which exceptions under 49 CFR 173.153 are allowed. This includes 1,1,1-trichloroethane, trichloroethylene, trichlorobenzene, PCB transformer oil, and many of the other poisonous materials commonly encountered.

3.6.1.3. CERCLA Reportable Quantities — DOT Class 9

Table 3-2 Column 3 lists packaging procedures for substances (liquids and solids) where the waste material is not otherwise classified as a DOT Hazardous Material because of hazardous properties *and* for which the entry in Column 8a of 49 CFR 172.101 Table is 155. For the shipment of larger quantities of EPA hazardous waste and DOT Class 9 hazardous substances where the quantity of material in each container *exceeds* the CERCLA RQ and no other DOT Hazardous Material classification applies, the following packaging requirements apply:

- Label each container with a separate container number
- Seal each drum or pail with a Security Seal
- Prepare one COC record for each group of containers that is being shipped at the same time to the same destination. List the container numbers on the COC record.

These shipments may include EPA Hazardous Waste in 5-gallon cans and 55-gallon drums. Most DOT containers are approved. The list of approved containers for packing Groups II and III Class 9 Hazardous Substances are listed in §173.203 for liquids and §173.213 for solids. These lists include steel, aluminum, plastic and fiber drums (solids only). Quantity limitations are shown in 49 CFR 172.101, Column 9.

Table 3-1. Packaging by Common Carrier

	By Common Carrier	
Instructions	**Non-hazardous Samples**	**Hazardous Samples**
Secure sample container lids with strapping tape.	1*	1*
Mark the level of material in each sample container with a grease pencil.	2	2
Place each container in a clear plastic resealable food bag so that the sample container label can be read.		3
Place about ½ inch of inorganic cushioning material such as vermiculite in the bottom of a metal can.		4
Place each container in a separate can and fill the remaining volume of the can with an inorganic cushioning material such as vermiculite (do not use plastic foam cushioning material as it could dissolve if the sample container were to leak).		5
Close the can using three clips to secure the lid.		6
Write the sample number on the can lid. Indicate "This Side Up" by drawing an arrow on the can.		7
Put about 1 inch of cushioning material (e.g., vermiculite or plastic foam) in the bottom of a watertight metal or equivalent strength plastic shipping container. If the container is a cooler, seal the drain plug on the inside of the cooler with tape. Also line the inside of the container with a plastic bag.	3	8
Wrap glass bottles and jars in plastic bubble wrap.	4	
Place cans in the container and fill the remaining volume of the shipping container with packing material. Add ice bags if required.		9
Place the sample containers top-up in the shipping container. Arrange the sample containers so that glass containers are surrounded by plastic containers.	5	
Fill the void space around and on top of the sample containers with plastic bags filled with ice cubes or ice chips.	6	
Seal the COC record in a clear plastic resealable food bag and tape it securely to the inside of the shipping container lid.	7	10
Close and lock or latch the shipping container.	8	11

	By Common Carrier	
Instructions	**Non-hazardous Samples**	**Hazardous Samples**
If the shipping container used is a picnic cooler, use tape to seal the drain plug.	9	12
After acceptance by the shipper, tape the shipping container completely around with strapping tape at two locations. Secure the lid with tape. Do not cover any labels.		13
Place the laboratory address on the top of the shipping container.		14
For all hazardous shipments, complete shipper's hazardous material certification form.		15
Place a “This End Up” label on the lid and on all four sides of the shipping container.	10	16
Affix the signed and dated custody seals on the front right and back left of the shipping container. Cover the seals with wide, clear tape.	11	17

*Numbers indicate the instructions that must be followed.

Table 3-2. Packaging Not by Common Carrier

Instructions	Flammable Liquid	Poison DOT Class 6.1	DOT Class 9
Quantity limitations shipped by cargo aircraft			
Gross weight of package:	66 pounds	66 pounds	66 pounds
Total quantity of flammable liquid:	49 CFR 172.101 Table, Column 6b		
Maximum sample container size:	49 CFR 172.101 Table, Column 5 *or* The flash point of the liquid	*Liquids* – 4 liters (1 gallon) *Solids* – 5 kilograms (11 pounds)	*Liquids* – 4 liters (1 gallon) *Solids* – 5 kilograms (11 pounds)
Check the caps of all sample containers to assure that they are secure. Tape caps.	1[*]	1[*]	1[*]
Place each sample container in an individual 6-mL plastic bag and secure with a twist tie. The sample identification tag should be positioned to enable it to be read through the bag.	2	2	2
Place sample containers in paint cans in a manner that will prevent bottle breakage.	3	*Liquids:* 3	
Place vermiculite in the paint can around the samples. The amount of vermiculite used should be sufficient to absorb the sample if a sample container should break.	4		
Secure the lid to the paint can with can clips and label the outside of the can with the sample ID numbers and quantity.	5		
Wrap bubble wrap around each glass sample container and fix with tape.		*Solids:* 4	3
Package the paint cans in DOT boxes or cooler. Use additional packaging to secure cans.	6		
Seal the drain plug with tape on the inside and outside of the cooler and line		5	4

Instructions	**Flammable Liquid**	**Poison DOT Class 6.1**	**DOT Class 9**
the cooler with a plastic bag. Place the canned or bagged sample containers in the cooler. If plastic bottles are being used, alternate them with any glass container.			
Fill any voids in the cooler with additional packing material.	7	6	5
Place ice contained in bags on top of all sample containers within the cooler. Use as much ice as space will allow.	8	7	6
Place the COC record in a clear plastic resealable food bag and tape to the inside of the cooler lid. Label the outside of the cooler as containing the COC record.	9	8	7
Seal the cooler lid with clear tape or strapping tape. Affix security seals.	10	9	8

*Numbers indicate the instructions that must be followed.

3.6.2. Marking and Labeling

All samples *must be labeled* to prevent misidentification and should include the following information:

- Sample # or ID
- Date of collection
- Collector
- Analysis requested
- Preservative
- Sample location.

Sample labels must clearly link the sample to the field sheet or the COC record and must be written legibly and in permanent ink. In addition, all containers must be labeled and listed on the COC record.

Note: If a three-bottle set is used for VOAs, all three bottles must be labeled and listed on the COC record.

EPA TSCA regulations [40 CFR 761.40(e)] require that a PCB label be put on all containers whose surfaces are in direct contact with material that is over 50 parts per million (ppm) PCBs. This requirement applies to sample containers as well as pails, drums, and other containers that are in direct contact with the PCB material. The labeling requirement does not apply to containers in which PCB sample containers are shipped. Although the sample containers must be individually labeled, this requirement is not affected by the quantity of sample or whether the sample is classified as hazardous under RCRA or DOT regulations. For DOT Class 9 and EPA Hazardous Waste the following labeling requirements apply:

- If EPA Hazardous Waste Manifest is required:
    - Hazardous waste
        - liquid, NOS, NA3082
        - solid, NOS, NA3077
- If EPA Hazardous Waste Manifest *is not* required:
    - Environmentally hazardous substances
        - liquid, NOS, UN3082
        - solid, NOS, UN3077

OSHA’s Hazard Communication Standard requires all containers of hazardous materials coming in or out of a workplace to be labeled with the contents, appropriate hazard warnings, and the name and address of the manufacturer. OSHA does not specify a standard labeling method, but some commonly used ones are provided by National Fire Protection Association (NFPA), Hazardous Materials Identification System (HMIS), ANSI, and DOT.

3.6.3. Shipping Papers

Ship high hazard samples via overnight courier following the courier’s documentation requirements. A special airbill must be completed for each shipment. An EPA manifest must be prepared if the shipping container contains hazardous waste *unless* the samples are exempt. The Hazardous Waste Manifest must bear the handwritten signatures of the generator, transporter, and designated facility. A copy of the manifest must be kept for 3 years by the shipper. The shipping papers must contain the name, address, and handwritten signature of the shipper.

The shipping papers (and Hazardous Waste Manifest if used) must contain a 24-hour emergency response telephone number. This phone number must be monitored at all times while the hazardous material is in transportation, including storage incidental to transportation. The phone must be monitored by a person who is either knowledgeable of the hazards and characteristics of the hazardous material being shipped and has comprehensive emergency response and incident mitigation information for that material, or who has immediate access to a person who possesses such knowledge and information. The emergency response phone number must be entered on the shipping paper immediately following the description of the hazardous material or entered once on a shipping paper if the number applies to all of the hazardous materials and is indicated for emergency response information.

3.7. QA/QC Protocol

QC is a normal part of good field and laboratory practice. QC includes all of the procedures applied to data collection and generation activities to achieve and maintain the level of pre-established data quality. The desired level of data quality should be based on the intended use of the data. Therefore, the QC protocol should include all technical controls (e.g., sampling and analytical methods, use of field blanks, field duplicate samples, inclusion of performance testing or reference samples, statistical analysis, etc.). The controls start with the regulatory requirements of the data acquisition project and carry through to the ultimate data reporting and completion of all of the documentation of the use of these controls.

QA refers to the procedures used by management to assure that the QC is what is required and that it is being adhered to at any point in the project. QA constitutes the overview and monitoring processes designed to ensure that the quality of the data generated meets the desired levels as established by management. These controls include establishing DQOs based on the intended use of the data, the institution of procedures for formalizing planning documents prior to the initiation of data collection activities, and the use of audits to identify problems in both QC and QA.

The QA/QC protocol is specified in the SAP for each job that involves field sampling. QA/QC requirements are based on the level of data quality required for the project, and may address specific regulatory requirements. The purpose of a QA/QC protocol is to ensure the following:

- The laboratory receives samples that accurately represent the conditions existing at the sample site
- The results of the analysis are traceable to the specific sample location
- Compliance requirements are met.

The methods used to attain this protocol include training of personnel, providing detailed procedures for preparation, collection, marking and handling, packaging, packing, transfer of samples, and validation and verification of the administrative process and sampling techniques.

3.7.1. Decontamination of Sampling Equipment

The SAP should address the extent of decontamination and specify the procedures to prevent sample contamination. Sampling may be performed using separate laboratory cleaned equipment for each sample location. Procedure effectiveness should be checked for each matrix by submitting equipment decontamination blank samples to the laboratory for analysis.

Note: For specific information regarding the decontamination of field equipment, refer to *ASTM D5088-02, Standard Practices for Decontamination of Field Equipment Used at Waste Sites*. This standard describes the decontamination process for field equipment used in the sampling of soils, soil gas, sludges, surface water, and groundwater at waste sites. According to this standard, these practices are applicable only at sites where chemical (organic and inorganic) wastes are a concern, *not* for radiological, mixed (chemical and radiological), or biohazard sites.

3.7.2. Sample Container Cleanliness Requirements

Sample containers are a possible source of sample contamination. The SAP should specify the level of QC for sample containers. Pre-cleaned containers meeting EPA CERCLA cleanliness endurance criteria are available from several suppliers. If these containers are used, the serial number and QA batch number of each one should be recorded in the FLB/FN or on the field form. A review of the cleanliness should be made to ensure all parameters are checked to be below the detection limit of the contaminants to be tested for compliance. Some SDWA and CWA parameters may require laboratory cleaned containers proven to be below the limit of detection for the method.

Note: In no case should an effort be made in the field to decontaminate a sample container. If a container becomes contaminated, it should be replaced, with a note to that effect recorded in the FLB/FN.

3.7.3. Sample Container Type and Size Requirements

The types and sizes of sample containers to be filled for each sample will depend on method requirements and on QC requirements of the SAP. General sample container requirements are shown in Appendix B for different matrices and analytical parameters. Compliance with specific instructions in the SAP is mandatory. If specified sample containers are not available, permission must be obtained from the Program Manager in writing for the use of other sizes and types of sample containers.

3.7.4. Sample Preservation and Storage Requirements

Special preservation and storage requirements should be specified in the SAP to ensure that samples do not undergo chemical changes from the time they were collected until their analysis by the laboratory. General requirements are specified in Appendix B. The specific requirements of the SAP will govern.

The quality of the reagents, water and materials used for preservation should be verified to ensure these items do not invalidate the reported results. Chemicals used as preservatives may be traced by lot number and quality by maintaining a reagent record keeping system. The water and acid preservatives used for trip and field blanks may be checked prior to use in the field and lot controlled to ensure no contamination is present prior to the material leaving the laboratory.

3.7.5. Sample Holding Time Limits

Even with preservation and special storage procedures, the composition of samples can change over time. The holding time for samples is the time from collection to laboratory preparation or analysis. Holding time limits summarized in Appendix B are method and program requirements. Site-specific holding times specified in the SAP take precedence.

3.7.6. Laboratory and Field Analytical Procedures

Laboratory analytical procedures for each parameter are specified based on the compliance limits, permit limits and data needs stated in the SAP. The SAP or COC record indicates to the laboratory which sample containers are to be analyzed for what parameters and specifies the analytical methods. Based on the DQOs, field testing may require the same level of QC as laboratory testing, and the procedures specified in the field sampling or test plan must be followed exactly. Any deviations from established test procedures must be entered in the FLB/FN or on the field form and the Program Manager must be informed immediately of sample numbers affected.

3.7.7. QC Samples

Field QC samples are prepared and analyzed to determine whether test samples have become accidentally contaminated, check on the repeatability of the method, and ensure the samples are representative of the site or matrix sampled. A number of different QC samples may be specified. Each of the QC samples checks for a potential problem that can affect data reliability. The recommended frequency for each type of QC sample is summarized in Appendix C.

3.7.7.1. Test Sample

The test sample consists of one or more sample containers filled with material collected at one sampling point within a stated time. Several sampling containers may be required if material collected for analysis for different parameters must be preserved differently or sent to different laboratories. For a specific test sample, all containers are designated by the same sample location number, but may have different sample container numbers or designations to indicate variations made to the samples.

3.7.7.2. Field Duplicates and Split Samples

Field duplicate samples are two separate samples taken from the same source and are used to determine data repeatability based on field sampling and laboratory analysis procedures. Field duplicate samples are as follows:

- Assigned different container numbers
- Specified in the FLB/FN or on the field form
- Distinguished from the test samples on the COC record or field records
- Often submitted blind as to designation so the laboratory data assures objectivity.

Exception: Each test sample collected for a specific organic analysis may consist of two or more containers filled with the same material; these may be given different container numbers but are designated as the same sample on the COC record. Only one sample container will be analyzed; the other being saved as a backup in case the laboratory must repeat the extraction and/or analysis. Duplicate samples for analysis consist of sets of two containers, with each pair of containers being designated on the COC record.

Field duplicate samples may be submitted to one laboratory for analysis for the same parameters. The comparability of the results provides information on the repeatability of the field sampling and laboratory analysis procedures.

The containers may be submitted to different laboratories for identical analyses to obtain information on inter-laboratory repeatability of field sampling and laboratory analysis procedures. This is a split sample.

Sample heterogeneity may cause major problems with the representativeness of field duplicate or split samples of soil/sediment matrices. Proper sample homogenization in the field will significantly improve the repeatability of the field sampling procedure. (Gy P. 1993, *Sampling for Analytical Purposes*, Wiley, West Sussex. Pitard F. F. 1993, *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sample Correctness and Statistical Process Control,* Books Britain, London.)

Typically, both field duplicates and split samples will be collected at a rate of 10% of field samples or at a minimum of one, per analyte, matrix, and sampling technique. More duplicates and split samples may be collected depending on the data quality needs.

3.7.7.3. Equipment Decontamination Blanks

Equipment decontamination blanks, or rinsate blanks, provide information on the levels of cross-contamination resulting from field or laboratory sample preparation actions. These blanks are specified in the SAP and on field sampling forms, and are prepared in the field. An equipment decontamination blank is usually reagent or deionized water that is free of the analyte of interest and is transported to the site, opened in the field, and poured over or through the sample collection device, collected in a sample container, and returned to the laboratory and analyzed. This serves as a check on sampling device cleanliness. For example:

- **Field Groundwater Equipment Decontamination Blank for Metals Analysis.** Handled by the bailer, use ASTM Type II water, or better. Filter, place in a sample container, and preserve using the same procedures as for the test and duplicate samples.
- **Soil Sampling Equipment Decontamination Blank for Semivolatile Organics.** Rinse the field equipment prior to its use and collect the rinsate for analysis.
- **PCB Wipe Sample Equipment Decontamination Blank.** Use a wipe pad to wipe the sampling template in the same way the pad is handled during the actual wipe sampling of a surface.

One equipment decontamination blank is collected for each type of equipment used during the day or sampling event. Equipment decontamination blanks are assigned container numbers from the same sequence as the test samples, and may not be distinguished from the test samples on the COC record. More blanks may be collected depending on the data quality needs.

3.7.7.4. Field Blanks

Field blanks are prepared and analyzed to check cleanliness of sample containers, environmental contamination, and purity of reagents or solvents used in the field. A sample container is filled with laboratory ASTM Type I or II water, preserved, shipped to the field with clean sample containers, opened in the field to exposure to ambient field air for a time compatible to field sampling process, and is closed and submitted for analysis using the same parameters as the test sample. The reported results will indicate the presence of contamination. Field blanks are most often used when measuring for volatile analytes.

3.7.7.5. Trip Blanks

A trip blank is used with VOA analysis of water. A blank may consist of two 40-milliliter VOA vials filled at the laboratory with laboratory ASTM Type I or II water, transported to the sampling site and returned to the laboratory without being opened. This serves as a check on sample contamination during sample transport and shipping.

Note: The caps used on VOA vials have Teflon®-lined septa. The Teflon® side of the silicone septum should face the sample. Prior to closing a vial, make sure there is no soil particle or dirt on the sealing surface of the VOA vial to prevent leaks. If a high concentration of volatile chemicals is present in the air in a shipping container, these chemicals can pass through the septum and contaminate the sample.

A trip blank is included in each shipping container used to ship VOA water samples. One VOA trip blank (two vials) is submitted to the laboratory in each cooler or per sampling event. The frequency of collection for trip blanks is specified in the SAP and is based on the data quality needs. Trip blanks are assigned container numbers from the same sequence used for the test samples, and are not designated as blanks on the COC record.

3.7.7.6. Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

Project or compliance QC procedures require that the laboratory spike a portion of the matrix with a predetermined quantity of analyte(s) prior to sample extraction/digestion and analysis. The frequency of performing an MS is dependent on the data quality needs and method requirements.

A spiked sample is processed and analyzed in the same manner as the sample. The result of the analysis of the spike compared with the non-spike sample indicates the ability of the test procedures to recover the analyte from the matrix, and provides a measure of the performance of the analytical method executed by the laboratory.

For an MSD, a second portion of the matrix is spiked, and the recovery of the MSD can be compared with the recovery of the MS.

Depending on the matrix and analysis, additional sample containers may be specified to provide enough material for this laboratory procedure. These sample containers are assigned container numbers from the same sequence as the test samples and are designated MS/MSD materials on the COC record.

The MS/MSD samples are commonly used in CERCLA testing, but are not commonly used in CWA or SDWA testing. MSs are routinely performed by the laboratory as part of its internal QC on randomly chosen samples. If MS data is required for SDWA or CWA reporting requirements, a request must be made to the laboratory to ensure the MS is performed and reported on the appropriate sample. The sample selected for MSs should have the same or similar matrix as the field samples' but without high levels of target analytes.

3.7.8. Field Audits

The SAP will specify who will conduct field audits, along with their frequency and procedures. QA/QC procedures of the sample collection effort must identify and determine the magnitude of error associated with the contamination introduced through the sample collection effort. Audits are perhaps the most effective tool to ensure that the sampling is done correctly. The two factors most likely to influence the magnitude of the sample collection error are collection methods and frequency of sampling.

In general, a field sampling audit provides an independent outside check on the following:

- **Field Records**
    - COC records
    - Sample container labels
    - FLBs or field forms
    - Personnel training records
- **Sampling Procedures**
    - Equipment
    - Sample containers
    - Accuracy of sample location descriptions
    - Comparability of field sampling techniques
    - Collection and preparation of QC samples
    - Sample preservation
    - Equipment decontamination
    - Contaminated waste storage and disposal

  - Sample packing, storage, security, and transportation
  - Shipping containers, including use of custody seals (if applicable).

3.8. Generic Sampling Equipment List

Equipment specific to each type of media is found at the end of the related chapters. The following is a generic sampling equipment list:

- Map of sampling location(s)
- Sampling SOP
- FLB or field form
- Pens
- Containers
- Preservatives
- Labels
- Markers
- Coolers
- Ice
- Packing material
- Packaging tape
- COC form
- Custody seals (if required)
- Decontamination storage containers, equipment, and materials
- Personal safety equipment, safety test equipment
- Field screening or testing equipment, standards, reagents, and SOP
- Testing field forms or logbooks
- Laboratory instructions (if different from custody form).

Chapter 4. Soil Sampling

4.1. Purpose

This chapter identifies the major steps that are required and technical areas that will be encountered when soil sampling. The scope of the sampling program is defined, as well as procedures that will guide the sampler in conducting soil sampling.

4.2. Scope

The characterization of soil and soil contamination requires a collection of samples from identifiable locations. Soil sampling programs can accomplish the following:

- Determine site soil characteristics (presence and location of different types of soil, subsoil, and bedrock)
- Define the extent, depth, type, and severity of soil contamination
- Determine soil "cleanliness" during procedures such as UST removal, or PCB transformer operation
- Soil sampling programs also can provide for background monitoring at treatment, storage, and disposal facility (TSDF) or hazardous waste handling areas.

4.3. Project Planning

This section provides an overview of the planning process for conducting soil sampling. Included is a discussion of sampling strategy, development of a site-specific SAP, and QA considerations.

Before soil sampling can begin, clear and concise objectives regarding the intent of the sampling program need to be developed. The objectives provide the framework for developing a sampling strategy for the site and preparing the SAP. Once the objectives are established, a sampling strategy can be developed.

4.3.1. Sampling Strategy

The sampling strategy is designed around the objectives of the project and should begin with a review of the conceptual site model. The model takes into consideration historical site use, known or suspected pollutant releases, and the geology of the area. The conceptual site model is dynamic and is constantly revised as new information is collected and processed. The model focuses on contaminant fate and transport processes, the control of geologic materials on the contaminant pathways (e.g., depositional environments, geologic structure), the types of contaminants present (e.g., hydrophobic versus hydrophilic), and the processes that influence the concentrations of contaminants present (e.g., dilution, biodegradation, dispersion).

The detail of the conceptual site model will depend greatly on the availability of information including historical site uses, native soil type, areas where fill materials may be present (e.g., utility trenches), and the direction of surface water flow. Specifically, the conceptual site model should describe:

- Physical characteristics of the site, including locations of buildings, paved areas, subsurface utilities, overhead utilities, significant topographical changes (e.g., steep banks), exposed bedrock, standing water, stained soils, areas of depressed vegetation, equipment maintenance and storage areas, manufacturing locations, waste disposal areas, and expected depth to groundwater
- Types of contaminants to be sampled (e.g., volatile organic chemicals (VOCs), semi-VOCs, metals) and factors that could bias sampling results (e.g., organic contaminants that are tightly bound to soil particles)
- Lateral and vertical distribution of contamination (e.g., contaminants distributed throughout an entire unit being monitored versus localized distribution controlled by small-scale features).

The sampling strategy and the information gathered from the conceptual site model will become the framework for development of the site-specific SAP. Key issues related to the conceptual site model are presented in more detail in the following subsections.

4.3.1.1. Types of Contaminants

The sampler or Site Manager may be able to predict the types of soil contaminants present and the duration of contamination by consulting site historical records and information on previous manufacturing practices, fuel or chemical usage, and any reported spills. This information can be used to determine sample collection locations. For instance, heavy metals such as oxidized lead or organic chemicals such as pesticides which are relatively insoluble in water may be limited to the top few inches of soil and will not have spread far from their source in undisturbed sites. However, if sites are affected by soil erosion or construction activities, the contaminants may have moved from their source, and sampling program planners should account for these changes in site conditions. The extent to which the underlying groundwater is contaminated also would be a function of the solubility of the organic liquid in the water. Dense non-aqueous liquids, such as chlorinated solvents (e.g., trichloroethylene, tetrachloroethylene, carbon tetrachloride) or heavier chlorinated organic liquids (e.g., chlorinated benzenes, PCBs), can permeate the subsurface until they reach a confining layer, such as clay or bedrock.

For soil contaminants such as soluble metals found in metal plating wastes or from spills of concentrated organic chemicals (e.g., gasoline, aviation fuel, solvents, and transformer oil), the presence and extent of contamination may not be easily predictable due to a number of factors. Soluble metals and organic liquids often are carried through soil by percolating rain water. The extent of contaminant movement is affected by a number of factors, including:

- **Density of Organic Liquids.** Light organic liquids (e.g., gasoline, diesel fuel, and waste oils) will settle through soils that are not saturated with water until they reach a confining surface such as clay, bedrock, or groundwater. It is not unusual to find a concentrated layer of organic liquid moving along the surface of a tilted clay layer or forming a pool or lens on the groundwater. Therefore, the extent to which the underlying groundwater is contaminated, is a function of the solubility of the organic liquid in the water. Dense, non-aqueous liquids, such as chlorinated solvents (e.g., trichloroethylene, tetrachloroethylene, carbon tetrachloride) or heavier chlorinated organic liquids (e.g., chlorinated benzenes, PCBs) can continue to sink through groundwater until they reach a confining layer, such as clay or bedrock. These chemicals can flow along tilted surfaces or pool in the cracks in bedrock and then slowly leach into groundwater.
- **Susceptibility to Biodegradation.** Microorganisms in the soil can degrade many contaminants. However, for contaminants like fuels or other petroleum distillate products, the microorganisms require oxygen and generally are not active at depths of more than 1 foot. Therefore, the surface soils may be relatively uncontaminated by these organic chemicals due to biodegradation, while deeper soils may be contaminated due to the lack of oxygen needed for biodegradation.

4.3.1.2. Type of Soil

Soil type can have a significant impact on the mobility of pollutants. For example, clay layers create relatively impermeable barriers, retarding groundwater flow and in some instances preventing surface contaminants from affecting groundwater. However, clay particles also can absorb certain types of hazardous chemicals, resulting in increased pollutant concentrations in localized areas. Organic liquids can form pools in the low reaches of clay layers, or can flow down the surface of tilted clay layers.

Soils that contain large amounts of organic material, such as peat, can absorb and concentrate hazardous chemicals. A layer of organic soil, therefore, may be more contaminated than the soil layers above and below it.

Porous non-organic soils, such as sand or gravel, do not absorb pollutants and allow for rapid surface and groundwater movement. In such soils, PCBs from spilled transformer fluid and from buried electrical capacitors have been found several hundred feet from the source of the contamination. Gasoline from leaking USTs can be carried hundreds of feet by moving groundwater.

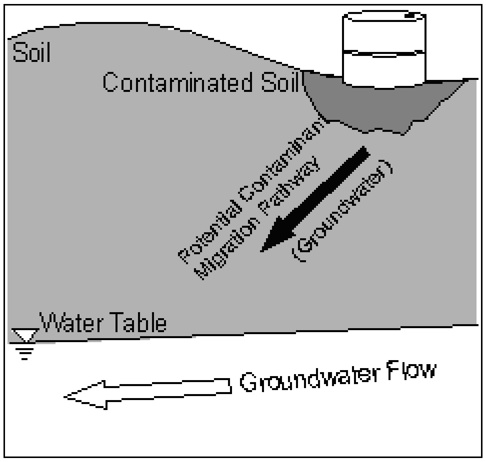
Bedrock may provide a confining layer beneath groundwater. Fractured bedrock, however, may provide channels for the movement of groundwater and heavy organics or low spots for the accumulation of pools of heavy organic liquids.

Soil characteristics such as pH, can change the chemistry of the surrounding water, which in turn can affect the solubility of some pollutants, including heavy metals. For instance, acidic stormwater can dissolve metals on the ground surface. However, as the dissolved metals pass through the soil column, they may encounter areas with elevated soil pH (e.g., limestone) that cause the metals to become insoluble precipitates again.

4.3.1.3. Presence of Groundwater

Moving groundwater can carry hazardous contaminants for considerable distances; see Figure 4-1.

Figure 4-1. Contaminated Groundwater



The rate at which groundwater will cause contaminants to spread is affected by both the speed at which the groundwater moves through the soil and the extent to which the soil absorbs the contaminants. One of the most serious effects of accidental spills and improper disposal of hazardous chemicals is contamination that precludes the use of a groundwater source for drinking water.

4.3.2. Site-Specific SAP

A soil sampling program should collect soil samples at a specific locations and depths that are representative of the site. Procedures that result in the collection of an undisturbed soil column will yield the most representative soil samples. The development of a site-specific SAP is the first step toward collecting a representative soil sample (see Chapter 2 for a detailed discussion of SAP development applicable to all sampling programs). Each SAP is an instruction manual for field personnel, and should be built around the objectives and sampling strategy. The discussion below focuses specifically on SAP development for soil sampling programs.

Ideally, the SAP should be based on knowledge of which contaminants are likely to be present and how the distribution of the contaminants may be affected by the soil characteristics of the site. The purpose of the fieldwork also may be to define the soil characteristics and obtain chemical data for determining compliance with federal, state, or local requirements. It is possible for soil sampling to be conducted in a number of phases, with the field observations and field test results providing data to guide subsequent work. If knowledge of contaminants and site soil characteristics is limited, then dynamic sampling techniques and field measurements can be used to locate sampling points.

Dynamic sampling relies on individuals in the field to interpret field data as it becomes available and revise the sampling strategy accordingly. Dynamic sampling can quickly provide significant amounts of information needed to develop the conceptual model for the site. Dynamic sampling in many instances, however, is considered a first step toward developing a more focused soil sampling program for a particular area within the site.

The SAP should consider a variety of factors, including, but not limited to, the physical features of the site (e.g., locations of buildings, tanks, buried utilities, roadways), the types of contaminants and their potential mobility and biodegradability, soil types, the accessibility of potential sampling locations, the size of equipment needed to collect samples at depth, suspected pollutants, field screening requirements, DQOs, analytical methods and detection limits, soil collection methods, sample handling procedures (e.g., preservation requirements, COC), containment and disposal procedures for contaminated soils generated during sampling activities, and safety.

The SAP cannot be implemented effectively if field personnel do not understand its contents. Many times the primary reason that sampling events are problematic is that the person who prepared the SAP is not the person in the field. One method that alleviates this problem is assigning a Sampling Team Leader who is responsible for all activities in the field. The Sampling Team Leader will work closely with those individuals preparing the SAP to understand fully the objectives of the program. The Sampling Team Leader then will be responsible for all individuals in the field. He or she also is responsible for providing additional training to field personnel, if required, before fieldwork begins.

4.3.3. QA

Soil sampling programs encompass a variety of information sources and include both primary and secondary data collection. These data sources are used to continuously update the conceptual site model and allow site managers to make decisions regarding further investigation, additional monitoring, remediation alternatives, or site closure.

The level of data quality for each soil sampling program, and for each specific sampling event, depends on the intended use of the data. For example, the level of QA/QC needed to estimate the volume of soil that may need to be removed for a corrective action might be different than for samples needed for site closure. It is important to remember, regardless of the level of QA/QC for any soil sampling event, that sample integrity must be maintained during sample collection. Laboratory analysis, no matter how sophisticated, is defensible only if the sample supplied to the analyst has retained its integrity.

To ensure quality data, site managers and those responsible for data integrity should begin with a systematic planning process that helps define the DQOs. The DQOs clarify the study objective, define the most appropriate type of data to collect, determine the most appropriate conditions for data collection, and specify tolerable error limits that will be used as the basis for program decision making. Detailed information on QA/QC, including the systematic planning process, development of DQOs, and preparation of SAPs, is available in Chapter 2 of this handbook.

Some specific QA/QC issues related to soil sampling programs include equipment specifications, equipment decontamination, and QC samples. Each of these is discussed briefly in the following sections.

4.3.3.1. Secondary Data

Secondary data are those data collected in addition to sampling data. For a soil sampling program, secondary data may include items such as historical records regarding site use, spills, and corrective actions on neighboring properties, current and historical subsurface utility maps, locations of previous above-ground and below-ground structures as well as possible disposal areas and areas containing fill materials or other antidotal agents. These data aid the Site Manager in selecting soil sampling locations or determining possible pollutants.

For secondary data, acceptance criteria are used instead of the measurement performance criteria typically used for laboratory data. In general, acceptance criteria are used to assess secondary data adequacy and evaluate uncertainty in the results derived from the use of secondary data sources. For example, the Project Manager for the soil sampling program may require interviews of former employees to validate the location of disposal areas found on historical site maps. In this case, the acceptance criteria are the verbal confirmation of the disposal areas by the former employees. The SAP for each soil sampling program will explain the acceptance criteria for determining which sources of data are sufficient to support the project objectives. See Chapter 2 for more information.

4.3.3.2. Equipment Specifications for Quality Control

The selection of appropriate materials for soil sampling equipment is critical to ensuring data quality. The materials that come into contact with the sample are as critical as the composition of the laboratory sample containers. Selecting the appropriate materials for sampling equipment should be based on the type of expected pollutants. It is important that the sample equipment not add or remove target pollutants. For example, if collecting samples for analysis of low-level metals, carbon steel sampling equipment should not be used. If samples are being collected for low-level organic constituents, plastic sampling equipment should be avoided because plastics have the potential to absorb organic pollutants.

The recommended materials for hand augers, split spoons, trowels, and other sampling devices are as follows: polytetrafluoroethylene (PTFE) (Teflon®), stainless steel, polypropylene, linear polyethylene, polyvinyl chloride (PVC), Viton®, and conventional polyethylene. Because soil sampling typically requires more rugged equipment compared with other types of sampling, stainless steel equipment is typically the material of choice.

4.3.3.3. Equipment Decontamination

Decontamination of existing and new equipment is required prior to use in the field. Section 4.9 describes decontamination procedures for various types of soil sampling equipment. Decontamination procedures must be followed and documented to prevent cross-contamination between sites and within the site. Rinsate blanks may be collected at the start and end of the sampling event to determine the cleanliness of the sampling devices and evaluate the cleaning techniques used in the field. Decontamination procedures for all equipment that contacts soils must be included in the SAP.

4.3.3.4. Quality Control Samples

Field duplicates or splits are collected in the field in double the number of bottles required for the regular sample. A duplicate sample is a sample collected concurrently, under comparable conditions, with a first sample. Duplicate samples are QC samples that are used to assess data repeatability based on field conditions. Duplicate samples provide a total precision of field sampling precision and lab analysis. Duplicate samples should not be identified as duplicates to the laboratory. Split samples are two or more representative portions taken from one sample in the field or in the laboratory and analyzed by different laboratories. Split samples are QC samples that are used to assess analytical variability and comparability. Samples sent to the same laboratory are duplicates, and samples sent to a different laboratory are splits. Split samples shall be field homogenized. To ensure that split samples are representative, the soil sample collection must follow Pierre Gy’s *Sampling Theory and Sampling Practices*. However, for VOC samples, split samples shall be collected as field duplicates to minimize loss of volatile components.

Procedures for obtaining split soil samples are included in Section 4.7.4.3.

4.4. Hazards and Safety Precautions

The HASP should address all anticipated hazards for each task. The following discussion contains common (and commonly overlooked) hazards associated with soil sampling activities. Refer to Chapter 2 for references to specific hazard identification techniques and detailed control measures.

4.4.1. General Safety

Some of the general safety hazards associated with soil sampling programs include the following:

- Moving sampling equipment and containers across rough terrain either by hand or by driving off road
- Use of heavy equipment such as drill rigs and power augers
- Conflicts with other uses of the area (e.g., traffic conflicts when sampling on or next to roads)
- Contact with overhead power lines when using a drill rig or a backhoe to gain access to subsurface soil.

4.4.2. Subsurface Hazards

Soil sampling is an intrusive activity that could expose sampling personnel to unidentified subsurface hazards, as discussed below.

4.4.2.1. Buried Munitions

If the probability of encountering buried munitions during the sampling program is moderate to high, a Required Explosives Safety Submission (RESS) waiver from the relevant explosives safety authority may be required prior to disturbing the site. In addition, a qualified explosives safety expert will be required to accompany the investigation team. See DoD TP18 for the minimum qualifications for unexploded ordnance technicians and personnel. The site should be checked by qualified personnel with ground-penetrating radar and/or metal detectors before any samples are collected. Only trained and certified personnel should handle or sample explosive or suspected explosive materials.

4.4.2.2. Buried Utilities

The use of augers and drill rigs to gain access to subsurface soil can damage buried utilities, including electrical and telephone lines and gas, steam, water, and industrial pipelines. Facility records should be reviewed carefully to identify any buried utilities in the sampling area. The site should be checked with ground-penetrating radar and/or metal detectors if there is any question as to the presence of subsurface utilities.

4.4.2.3. Toxic Chemicals

Collecting soil samples poses the risk of exposure to hazardous chemicals. The following major exposure scenarios should be evaluated:

- **Skin Contact with Contaminated Soil.** Field sampling personnel should wear disposable boot covers and surgical gloves as a minimum, and should consider the use of disposable Tyvek® coveralls to prevent contamination of clothing. The sample custodian should wear appropriate PPE when handling samples.
- **Skin Contact with Contaminated Water.** Deep soil samples usually result in the requirement to handle wet soil that can drip and splash. Decontamination of sampling equipment also can result in splashes of contaminated water. Field sampling and decontamination personnel should wear waterproof rain gear or coated Tyvek® coveralls if there is a possibility of contact with contaminated water.
- **Ingestion of Contaminated Soil.** Ingestion can occur when food or drink is exposed to contaminated dust or is picked up with contaminated gloves. Field personnel should not eat, smoke, or chew gum or tobacco when working with contaminated soil. Drinking water should be kept in closed squirt bottles or should be stored outside the contaminated area.
- **Inhalation of Contaminated Dust.** Inhalation of contaminated dust may be a problem on dry windy days at sites where the surface soil is contaminated. Digging and drilling activities also may generate contaminated dust. Personnel should remain upwind of any source of dust. If the site is generally contaminated, then full-face, air-purifying respirators equipped with particulate filters should be worn.
- **Inhalation of Toxic Vapors.** The exposure of contaminated soil to ambient air may generate toxic vapors from the evaporation of VOCs. The HASP should establish air monitoring requirements for intrusive work and specify

the type of air monitoring equipment to be used as well as the criteria for the use of respirators. Either air-purifying respirators or supplied-air respirators may be required, depending on the toxicity of the chemicals present, the capabilities of the monitoring equipment, and the warning properties of the vapors.

- **Confined Space Hazards.** In general, pits more than 4 feet deep are considered to be confined spaces. However, any pit dug in contaminated soil could have the same hazards associated with confined spaces because flammable vapors or toxic gases, including hydrogen cyanide and hydrogen sulfide, could accumulate. All applicable confined space entry requirements should be imposed and detailed in the HASP.

WARNING: Personnel who enter into a confined space should be accompanied by an on-site standby unit at all times.

The following steps should be detailed in the HASP prior to entry into a confined space:

1. Ventilate the area thoroughly prior to entry.
2. Disconnect connecting pipelines.
3. Take air samples to verify the absence of flammable or other hazardous vapors and demonstrate the presence of adequate oxygen levels.
4. Provide entry and standby teams with proper training, respiratory protection, protective clothing, safety harnesses, and ropes.
5. Establish lifeline signals (e.g., tugging on a rope) prior to entry so that Field Sampling Personnel can communicate.
6. Maintain ladder(s) and other equipment in accordance with OSHA regulations.

4.4.3. Explosive Hazards

Due to the inherent dangers involved in sampling explosives and potential or suspected explosives, only those individuals who have been trained and certified in the proper handling of these materials should participate in sampling activities. Special considerations are required when sampling explosive wastes that are susceptible to shock, friction, electromagnetic radiation, electrostatic discharge, sparks, flames, elevated or freezing temperatures, moisture, or sunlight. Failure to handle explosives correctly could result in damage to property, injury, or loss of life. For explosives safety and handling requirements, refer to DoD 6055.09-M, *DoD Ammunition and Explosives Safety Standards*. For Navy projects, also refer to NAVSEA OP5, *Volume 1, Ammunition and Explosives, Ashore Safety Regulations for Handling, Storing, Production, Renovation, and Shipping*.

General explosives safety considerations include wearing PPE such as flameproof clothing, caps, safety goggles or face shields, conductive shoes, and respirators, where appropriate. Only non-sparking tools should be used. Electrical grounding may be necessary in some cases. Sampling of the smallest amount necessary to perform testing is recommended. Specific precautions are material-dependent. It is imperative that sampling personnel have a thorough knowledge of the characteristic dangers and safety requirements for individual explosives materials.

4.5. Principles of Sample Collection

The collection of quality soil sampling data requires documentation of site surface conditions (e.g., locations of buildings, pavement, standing water, seeps, sediment runoff, discolored soil) and subsurface conditions such as depth of various layers of soil, depth to groundwater and bedrock at different locations. The chemical data obtained from the analysis of soil samples is useful only within the context of these site conditions, so it is imperative that the exact location of each sample be documented and the soil samples accurately represent the conditions at the site.

Soil samples must represent field conditions and address the data needs of the program. For instance:

- In determining whether the contamination of surface soil presents a hazard by skin contact or ingestion, the samples should consist of soil from the surface to a depth of only 2 inches, and the soil should be sieved to eliminate stones larger than 2 mm in diameter.

- EPA cleanup requirements for soil contaminated with PCBs are based on the concentration of PCBs in the soil, including rocks. The samples taken to define the location of PCB-contaminated soil and the appropriate remediation criteria should not eliminate rocks, because the cleanup standards are based on the maximum allowable average concentration of PCBs in the remaining soil.
- To determine whether excavated soil is classified as a hazardous material under RCRA, soil samples should be representative and include rocks and hard chunks. The extraction procedure specified by EPA may require that the lab screen the soil to eliminate rocks larger than 3/8 inch in diameter, but the criteria are too complicated to allow a decision to be made in the field as to whether the soil should be screened when it is collected.
- Special sampling procedures must be used when collecting soil samples that will be analyzed for VOCs because these chemicals rapidly evaporate from soil once exposed to the air. Unless precautions are taken to minimize this evaporation, the samples will not be representative of the actual level of contamination of the soil.

Another important data requirement from any field sample is the documentation of the exact location where the sample was collected. Stakes or flags should be installed to indicate the locations of soil samples. However, because sites probably will be disturbed by subsequent construction work, it also is important that the location of each sample be measured with reference to permanent features such as survey monuments, measurements to a reference point, or latitude and longitude location. For soil sampling programs, it is generally acceptable for the survey to define each sample location to an accuracy of 1 foot horizontally and a few inches vertically. Unless the area to be sampled is flat, an initial survey should be made to define the surface contour. Measuring the depth from the surface at which a sample is taken and at which different soil layers and groundwater are encountered is insufficient, unless the elevation of the surface at that point is known and will remain undisturbed.

The final important data requirement is the physical characteristics of the site, including the surface soils, subsurface geology and soil characteristics. The physical characteristics of surface soils help define the initial locations for soil borings and aid in selecting the appropriate laboratory analyses for future soil samples. The following locations and physical characteristics should be noted and included on a site map:

- Erosion patterns and accumulated runoff sediments
- Equipment or activities, including above-ground and underground storage tanks, chemical storage buildings, outdoor areas where servicing of automotive or mechanical equipment has occurred, raw material storage areas, locations of lagoons, waste pits and fire pits, and locations where trash is either disposed or stored temporarily, such as in dumpster areas
- Discolored soil
- Areas covered by impermeable material, such as pavement and buildings
- Areas of distressed vegetation
- Exposed bedrock
- Standing water
- Groundwater seeps.

Subsurface geology and soil characteristics are typically determined during the site investigation phase of a soil sampling program and are used to predict pollutant movement and develop alternatives for any potential remedial actions that may be required. Subsurface geology and soil characteristics data include the following:

- Type of soil as a function of depth at each sample location (e.g., layers of clay, peat, sand, silt, shale, layers having different colors or the presence of large stones)

- Soil moisture at different depths, and the depth at which the soil becomes saturated with water (i.e., depth to groundwater)
- Presence of organic vapors in soils at various depths.

Field sampling personnel often are in the best position to detect areas of suspicion and, therefore, are critical to the success of the sampling program. Though sampling techniques may be sophisticated, they should not be relied upon to replace the good judgment and common sense of sampling personnel in discerning the difference between routine and extreme case scenarios. Sampling personnel must be alert to their surroundings (e.g., unusual circumstances, odors, presence of dead animal or plant life in the area) while conducting the following sampling tasks:

- Collecting and recording visual and physical data
- Collecting soil samples, both surface and subsurface
- Adhering to field sampling and safety procedures
- Maintaining the COC record
- Preserving program integrity.

Techniques for preparing a site map and documenting field data and observations are discussed below.

4.5.1. Preparation of Site Map

Documentation of site conditions and sample locations requires a site map. The site map should be included with the SAP. The site map may be based on facility drawings but should be verified in the field and augmented with additional information on the drainage of rainwater and other surface conditions that affect the movement of contaminated soil. If the SAP does not include a site map, one should be prepared by Field Sampling Personnel based on the availability of facility drawings and field measurements.

Integrating a geographic information system (GIS) into a sampling program allows for creating maps in significantly less time compared with traditional computer aided design (CAD) procedures. GIS-based data analysis is quicker, easier, and more accurate compared with traditional CAD methods. GIS can be used to analyze site conditions such as depth to groundwater, saturated thickness, hydraulic conductivity, and soil type for selection of suitable sampling points as well as for analyzing relationships between sample locations, tracking sampling points, and relocating sampling points under changing site conditions.

The basic tools necessary for incorporating a GIS into a sampling program include a global positioning system (GPS) unit/receiver and a computer program such as the ESRI’s ArcGIS. The GPS receiver is used to locate predetermined sampling points in the field and/or record the location of sampling points as samples are collected. The GIS program can be used to analyze site conditions prior to the sampling event or map and analyze the collected data. Creating a GIS database at the early stages of the sampling process provides for an effective method for data management, analysis, and presentation. The use of a GIS can significantly streamline many aspects of a soil sampling program and facilitate the exchange of resultant data among stakeholders.

Site maps, whether prepared using GIS- or CAD-based systems, should include surface contour, surface information, and sampling locations, as discussed below.

4.5.1.1. Surface Contour

Unless the site is completely flat, it is important that the surface contour be indicated on the site map. The depth of soil samples is measured from the surface, but the surface reference can be lost due to excavation or filling activities at the site. If a contour map of the site is not available, it may be necessary to have one prepared by a survey team prior to sampling.

4.5.1.2. Surface Information

Sampling personnel should validate the site map before beginning any sampling activities, noting any discrepancies. The following surface information should be recorded if not already shown:

- Buildings or structures
- Paved areas
- Unpaved roads and parking areas
- Surface areas with different types of soils, including fill areas (e.g., gravel roads, clay, sand, peat)
- Standing water, both permanent (e.g., ponds and streams) and temporary (e.g., persistent puddles)
- Water seeps
- Water runoff patterns and accumulations of runoff sediments
- Exposed bedrock
- Discolored soil
- Stored materials, debris, and soil piles
- Vegetation, including distressed vegetation.

Photographs should be taken to document site surface features. Photography requirements should be established by the Program Manager to ensure compliance with the policy and regulations of the facility.

4.5.1.3. Documentation of Sampling Locations

The location of each sample and the designated sample numbers should be shown on the site map. Samples should be collected at the designated sampling points. Depth of samples from the surface should be recorded. If it becomes necessary to sample more than 1 foot from the required location due to interferences from trees, pavement, subsurface rocks, or buried utilities, the new sample location should be noted on the map, and the reason for moving the sampling location should be explained in the FLB/FN.

4.5.2. Preliminary Tests and Observations

A number of field tests and observations may be required to document sampling and subsurface conditions. Such tests may include weather conditions at the time of sampling, a description of the subsurface geology, and the presence of VOCs in soil vapor. Each of these preliminary tests and observations is described below.

4.5.2.1. Weather Conditions

The weather conditions at the time of sample collection should be noted in the FLB/FN. Weather data should include temperature, relative wind speed and direction, relative humidity, and the presence of rain or snow. Recent weather conditions at the site should be summarized, including recent rain events (how much and how recently) and freezing conditions (how cold and how deeply is the soil frozen).

4.5.2.2. Description of Soil Types and Soil Characteristics

The movement of groundwater and the transport of hazardous chemicals are strongly influenced by the presence and depths of different types of soil. Knowledge of subsurface soil characteristics is necessary to understand the dynamics of soil contamination.

The soil should be described by a professional geologist if this information will be used to support the computer modeling of groundwater flow. If this level of detail is not required, the soil layers and the soil samples should be described by Sampling Personnel.

Chapter 3 of EPA's *Description and Sampling of Contaminated Soils — A Field Pocket Guide* is a good source for the field description of soils. As necessary, the following soil features should be described as a function of the depth from the surface:

- **Color.** Soil colors should be determined with the use of a color chart, such as a Munsell chart
- **Mottles.** Blotches or spots of contrasting color interspersed with the dominant soil color
- **Soil Texture.** This is the amount of sand, silts, and clays in a soil
- **Particle Shape.** Shape of individual soil particles
- **Structure.** Shape of the natural soil aggregates

- **Consistency.** Degree of resistance to breaking or crushing (descriptions will vary with moisture condition)
- **Presence of Visible Organic Liquids.** Such as oil, gasoline, solvents or other
- **Horizon Thickness**. Layers of soil with distinct changes of the above features.

4.5.2.3. Soil Gas

Soil gas monitoring is primarily used to measure characteristics of the soil atmosphere as an indirect indicator of VOC contamination in soil or groundwater. The resulting information may be used to:

- Detect and monitor migration of volatile contamination of soils within the vadose zone
- Support the design of a soil vapor extraction system
- Assess the extent of groundwater volatile organics contamination
- Verify UST integrity
- Monitor for any subsurface discharges
- Target soil borings
- Aid in placing groundwater monitoring wells, extraction wells, or recovery trenches, etc.

The information also can be 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. For information on soil gas sampling as it relates to the evaluation of vapor intrusion, see the *DoD Vapor Intrusion Handbook*.

In many circumstances, soil gas sampling alone may not be adequate to determine the source and extent of contamination, and sampling of other environmental media may be necessary. Soil gas sampling should be designed to obtain all necessary information with a minimal expenditure of time and resources. Sampling strategies are developed by the project team to address project-specific data needs that are identified during the systematic planning process. Sampling strategies will vary based on the intended purpose of the soil gas survey. The overall effectiveness of a soil gas sampling method to depict subsurface conditions accurately is dependent on several factors. These factors include soil permeability and the presence of any underground obstacles or other features affecting sampling pathways, the nature and concentration of contamination and the presence of interfering chemical compounds, and infiltration moisture and barometric pressure changes.

Sampling considerations include site physical constraints, soil types and stratigraphy, and background information available. Multiple depth sampling should be considered when complex geologic settings are encountered. Sample locations also 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. Not all of these properties may be known in advance, but a review of any background information such as past sampling or well installation should be accomplished prior to soil gas sampling. If this information is not available, it may be beneficial to install a continuously cored boring to the proposed greatest depth of the soil gas investigation and examine the soil lithology. Low permeability materials may result in a lower flow rate for active sampling and passive sampling may be a better choice. It may also be necessary to install an increased number of sampling points in low permeability soil to achieve sufficient coverage, or reevaluate the need for that particular sampling location.

In addition, the properties of the chemical contaminants must also be considered, including volatility, solubility or immiscibility in water, and degradation potential. 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, vinyl chloride, etc.). Rainfall or wet soil conditions decrease the porosity of the soil and limit diffusion of contaminants. Soil gas sampling should not be performed after a rainfall event. Barometric pressure changes can introduce atmospheric air into the shallow vadose zone, so soil gas should not be sampled during the passing of a weather front. All of the factors listed above can affect the ability of contaminants to be properly sampled. In most cases, adjustments to the sampling strategy can be made to increase sampling effectiveness.

When soil gas sampling is used to optimize monitoring wells or soil boring locations or monitor the integrity of USTs, 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 the 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.

Soil gas samples may be generated in conjunction with an active or passive 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 source. The air within the system may be monitored real-time through a sample port with an appropriate detector (e.g., photoionization detector (PID) or flame ionization detector (FID)). Active soil gas systems may also be sampled with a gas-tight syringe, Tedlar® bags, glass bulbs, Summa® canisters, or other containment devices for subsequent onsite or off-site analyses.

The active soil gas sampling system may also be configured to collect a sorbent sample. In this type of system, the probe is connected to a collection device designed to extract and trap chemical contaminants from the soil gas stream by adsorption. The collection medium is dependent on the contaminant being collected and 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. Using sorbent media is especially useful when contamination is present that is below the instrument sensitivity capabilities for whole air samples, and pre-concentration of the contaminants onto the adsorbent can be accomplished by passing a large volume of air past the sorbent and trapping the contaminants of interest. Some sorbent media can also be used to segregate interfering compounds (e.g., reactive or oxygenated organic compounds) by treating or designing the medium so the interferences 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. Limitations in the collection of sorbent samples include interactions between the contaminants and the sorption medium, the release of artifacts during the desorption process, and susceptibility to 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 (a larger amount of soil gas is sampled more rapidly), the generation of concentration-based data (mass of contaminant per volume of air sampled) that can be used in a contaminant transport model or risk assessment, and the ability to tailor the survey as it progresses. Limitations of an active soil gas system include its increased cost compared with that of passive techniques because of the need for a vacuum source and additional collection equipment. Driven probes associated with active soil gas sampling 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.

A passive soil gas sampling system entails placing a collection device in the subsurface or on the surface of the ground. Most commonly, 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 gas 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. Advantages to the use of a passive soil gas sampling system include its nominal cost, its lower susceptibility to meteorological changes, and the increased ability to detect less volatile compounds when compared with active soil gas sampling. Limitations include an extended turnaround time for data, the need for more samples for thorough coverage because passive samplers generally sample a smaller volume of soil gas, the lack of concentration-based data, and it the inappropriateness for use with soil vapor extraction system design.

Soil gas samples may be analyzed onsite, or samples may be containerized and shipped to a laboratory for off-site chemical analyses. 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 (e.g., Tedlar® bags) in dark plastic garbage sacks, and keeping them 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.

The ASTM D5314 standard provides a guideline for setting up an active soil gas sampling system. The California Environmental Protection Agency’s Department of Toxic Substance Control also has comprehensive guidance on active soil gas sampling. Additional procedures may be necessary based on the manufacturer’s specifications.

Typically, the active soil gas sampling system involves sampling devices that are directly inserted into the soil to be sampled without drilling/excavation using static pressure, impacts, or vibration, or the sampling device is inserted into the soil ahead of an auger or drill bit if performing deep sampling or vertical profiling. 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. There must be a good seal between the probe and the earth’s surface to minimize pulling atmospheric air into the collector. A drop in vacuum or too low a vacuum may indicate system problems. Sample containers may include, gas-tight syringes, Tedlar® bags, Summa® canisters, etc., for a whole air sample. If using Summa® canisters to collect samples, ensure that the valves have remained closed during shipping from the laboratory and are fully functional. In some instances the laboratory will pre-set the flow rate into the canister, and sample volumes are calculated by the amount of time the valve is open, rather than by monitoring the gauge on the flow controller. Similarly, when collecting field replicate soil gas samples, proper flow splitters and controllers must be used to maintain the same flow rates to individual Summa® canisters. Additionally, the soil gas may be collected on an appropriate sorbent media and cartridge at a location between the probe and the vacuum pump, or analyzed by a real-time monitor such as a photo-ionization detector or FID. Appropriate decontamination procedures should be followed between each soil gas sample collected.

4.6. Methods of Accessing Soils

It is important that soil samples consist of reasonably undisturbed soil from the specified sampling depths. Access to the soil often requires drilling or excavation down to the specified depth. In some cases, it may be necessary to use a jackhammer to remove either concrete or pavement from the sampling locations. In instances where areas are grass-covered and no surface sample is required, several square feet of sod should be carefully cut away. A decontaminated stainless steel shovel should be used to carefully remove the turf so that it may be replaced when sampling is complete. The stainless steel shovel should be decontaminated between sampling locations. Turf also can be removed by hand from sample locations, provided that personnel wear clean nitrile or PVC gloves.

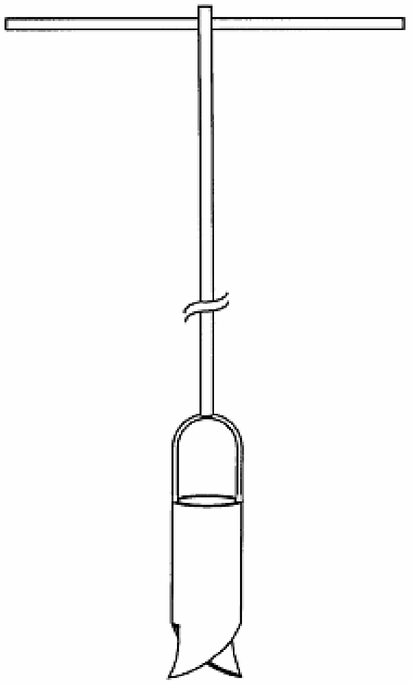
4.6.1. Surface Samples

Soil samples should be taken from undisturbed soil, not from material that is temporarily lying on the ground. Unless the SAP specifically requires the sampling of surface debris, remove organic debris such as leaves, and accumulated trash or demolition rubble, to expose the soil. The SAP should define the depth of surface samples. Specific state and local compliance programs may define surface samples as the first 3, 6, or 12 inches below the vegetation layer. Note in the FLB/FN how much and what kind of material is removed. Surface samples can be collected using stainless steel trowels, spoons, or bucket augers. A diagram of a typical bucket auger is provided in Figure 4-2.

4.6.2. Subsurface Samples

Subsurface samples should consist of undisturbed soil starting at the depth below the surface specified in the SAP. Subsurface samples should not contain material from different layers of soil. Collection of subsurface soil samples requires that the overlying soil be removed. The SAP should detail material separation and depths of subsurface sampling, including sample mixing, splitting, and representativeness.

Figure 4-2. Bucket Auger

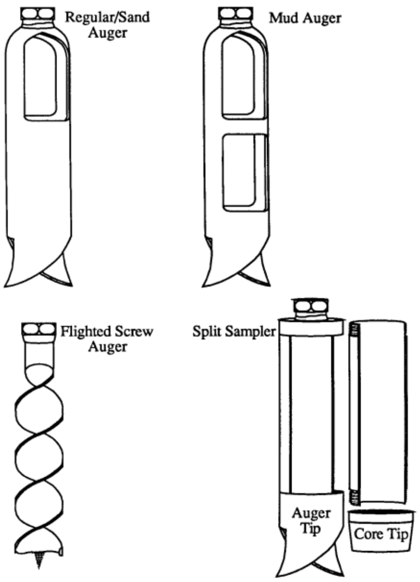


A variety of tools, ranging from handheld methods to larger fuel-powered methods, can be used to access unconsolidated soils. Consolidated soils can be difficult to sample because they are difficult to penetrate. Different drilling methods can be used to access consolidated soils. Truck-mounted drill rigs can gain access to soil at any reasonable depth and can penetrate hard layers. When used with a hollow-stem auger, a drill rig can retrieve reasonably undisturbed cores for classification purposes. The following subsections describe the various techniques used to access both unconsolidated and consolidated subsurface soils.

4.6.2.1. Augers

An auger is a hole-making tool that is screwed into the soil; see Figure 4-3. The auger is used to bore a hole to a desired sampling depth and is then withdrawn. In general, augers should be used only to gain access to the soil that is to be sampled. Augers churn the soil and destroy its structure, making soil classification more difficult and causing rapid release of volatile contaminants. Also, when augers are lowered into boreholes, they can scrape soil off the sides of the hole so samples collected from the bottom of the hole may be contaminated with soil from shallower levels.

Figure 4-3. Augers



Various types of augers are available. The major types of augers, their advantages, and their limitations are summarized in Table 4-1.

Table 4-1. Types of Augers

Sampling Device	Applications	Limitations
Screw Auger	Cohesive, soft or hard soils, or residues	Will not retain dry, loose, or granular material
Standard Bucket Auger	General soil or residue	May not retain dry, loose, or granular material
Sand Bucket Auger	Bit designed to retain dry, loose, or granular material, silt, sand, and gravel	Difficult to advance boring in cohesive soils
Mud Bucket Auger	Bit and bucket designed to wet silt and clay soil or residue	Will not retain dry, loose, or granular material
Dutch Auger	Designed specifically for wet, fibrous, or rooted soils (marshes)	
In situ Soil Recovery Auger	Collection of soil sample in reusable liners; closed top reduces contamination from caving sidewalls	Similar to standard bucket auger
Eijkelcamp Stony Soil Auger	Stony soils and asphalt	
Planer Auger	Clean out and flatten the bottom of predrilled holes	

A hand auger can be used to expose soil as deep as 4 feet. Gasoline-powered portable augers may be able to reach up to 12 feet depending on soil conditions, but are heavy, require two people to operate, and may contaminate samples when volatile organics are to be sampled.

The hollow-stem auger is commonly used in unconsolidated soils. The hollow-stem auger column rotates as it drills into the ground and is designed to push soil up and out of the borehole along the outside of the auger. The auger itself is driven either mechanically or by a hydraulicallypowered drill rig. A plug is placed through the auger to prevent soil from rising through the hollow portion of the stem. Samples are retrieved by retracting the plug and lowering the sample collection tube through the auger. Casings and screens for access holes can be placed in the hollow stem to prevent the borehole wall from collapsing and to ensure discrete interval samples. Casings also can be used to isolate near-surface contamination while drilling continues using a smaller diameter auger.

Auger Procedures

1. Clear the area to be sampled of any surface debris, such as twigs, rocks, or litter. It may be advisable to remove the first 3 – 6 inches of surface soil from an area 1 foot in diameter to prevent loose near-surface soil particles from falling down the hole.
2. Attach the auger bit to a drill rod extension, and then attach the drill rod to the T handle or the power unit.
3. Begin drilling, periodically removing, and depositing accumulated soils onto a plastic sheet spread near the hole. This practice prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods, facilitates refilling of the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the hole.

4.6.2.2. Direct-Push Techniques

Direct-push methods involve pushing a small-diameter hollow steel rod into the ground to a selected depth and can be used in most materials that can be augered or sampled with a split spoon. Direct-push techniques typically rely on a truck-mounted hydraulic ram to gain access to subsurface soils. The hydraulic ram pushes a closed-end ¾ – 2-inch diameter sampler into the soil, down to the sampling depth. The sampler contains a removable sampling tube and is capped with a retractable penetrating point. After the sampler is pushed to the desired depth, the penetrating point is retracted and the sampler is advanced to collect the soil sample inside the removable tube. This sampler can reach depths of 30 – 60 feet or more in sand or fairly loose soil, but has trouble penetrating clay and hard materials. As with any sampling device, hydraulic rams have both advantages and disadvantages.

The advantages of direct-push technology include the following:

- Retrieves relatively undisturbed soil cores up to 18 inches long. By collecting cores from different depths at closely adjacent locations, it is possible to develop a continuous core for soil classification purposes.
- Collects samples rapidly- one core sample can be collected every 10 minutes
- Minimizes the release of organic vapors, reducing personnel exposure hazards
- Generates little to no waste soil as compared to other drilling methods.

The primary disadvantages of direct-push technology include the following:

- Requires the rental of special equipment and trained operators
- Soil cores are only ½ inch in diameter, so several core samples may have to be obtained from a single sampling location to meet the laboratory sample quantity requirements.

Direct-push technology has rapidly become the method of choice for collection of subsurface soil samples due to its speed and low cost. There are currently numerous commercial environmental drilling companies located throughout the country that can provide direct-push soil sampling services.

4.6.2.3. Sonic Drilling

Sonic drilling is used for continuous sampling in unconsolidated and soft/fractured bedrock. The primary benefit of this technology is that very rapid drilling rates, combined with reduced volumes of secondary waste, are possible. Recent improvements in equipment design could lead to increased use in the future. A sonic rig uses an oscillator or head with eccentric weights driven by hydraulic motors to generate significant sinusoidal force in a rotating pipe drill. The frequency of vibration (generally between 50 and 120 cycles per second) of the drill bit or core barrel can be varied to allow for the optimum penetration of subsurface materials. A dual-string assembly allows for the advancement of casing along with the inner casing used to collect samples. Small amounts of air and water can be used to remove the material between the inner and outer casings. When a drill bit is used, most of the cuttings are forced into the borehole wall. A thin-wall or split-spoon sampler can be used to obtain continuous samples. Sonic drilling also is referred to as vibratory drilling and rotosonic drilling.

4.6.2.4. Cable Tool

Cable tool drilling is well suited for areas contaminated by hazardous substances because it does not use any circulation fluids that could potentially spread contamination. Auger drilling and sonic drilling are the only other drilling methods that do not use circulation fluids. Of these two, only sonic drilling has demonstrated the ability to contain contaminants as effectively as the cable tool.

There are two types of cable tool drilling methods used in the field.

- **Hard Tooling (Percussion Drilling)**. Best used below the water table in areas where unsaturated zone soils become consolidated.
- **Drive Barrel (Dry Drilling).** This technique is appropriate for relatively dry, unconsolidated soils, such as sand and gravel, often found in the unsaturated zone (i.e., the soil above the water table).

Cable tool drilling rigs operate by repeatedly lifting and dropping a heavy string of drilling tools attached to a cable into the borehole. The drilling string of a cable tool consists of five components:

1. Consolidated rock is broken or crushed into small fragments, and unconsolidated material is loosened by the drill bit or shoe.
2. The drilling stem connects the drill bit to the drilling jars.
3. The drilling jars are used to vibrate the drill bit free of the formation.
4. The swivel or rope socket connects the remaining drill tools to the cable.
5. The cable is strung over a pulley on the mast to the drill motor.

Hard tooling is the most common form of cable tool drilling and can be used in any formation, including basalt. The percussive action (producing a noise level of 53 – 115 decibels) of the drill bit crushes the formation. The cable is attached to an eccentric walking or spudding beam that also serves to mix the crushed or loosened particles with water (generally 10 – 20 gallons if no water is present in the formation) to form slurry at the bottom of the borehole. Periodically, the drilling string is removed, and the slurry is pulled from the borehole using a sand pump or bailer. The drive barrel method uses the cable tool rig to drive the drill casing into the soil. The soil is pushed inside of the casing and then collected in a split-spoon sampler or core barrel.

4.6.2.5. Test Pits

Under unusual circumstances, the excavation of a test pit may be useful in determining the depth and thickness of different types of soil or any apparent band of soil contamination. A backhoe can be used to remove sections of soil. This method probably is the most expensive method of accessing soils for sampling due to the relatively high cost of backhoe operation.

Samples should be collected from undisturbed portions of the walls or bottom of the pit, not from soil that is excavated from the pit. Before digging a test pit, four stakes should be driven into the ground far enough from the sample location that they will not be disturbed by the backhoe or other activities. The stakes should be located such that strings attached to opposite posts will intersect directly above the sampling location.

The following procedures are used for excavating test pits or trenches for soil sampling:

- Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities
- Review the site-specific HASP and ensure that all safety precautions, including installation of appropriate monitoring equipment, are taken as required
- Using the backhoe, excavate a trench approximately 3 feet wide and 1 foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than 5 feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.
- A shovel is used to remove a 1- to 2-inch layer of soil from the vertical face of the pit where sampling is to be done
- Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above and expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket if the soils in the bucket are representative of the target sampling location.
- Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can be backfilled with the removed soil material.

The advantages of excavating test pits include the following:

- **Visibility.** Provides the best information on the location of soil layers.
- **Versatility.** May be the only way to access subsurface soil where large rocks are present.

The disadvantages of excavating test pits include the following:

- **Safety**
    - The backhoe may have to be decontaminated before removing it from the site
    - Trench walls deeper than 3 feet may be unstable. Shoring may be required prior to having anyone enter the test pit to collect samples
    - Toxic or flammable vapors may accumulate in the test pit. Any entry into a test pit that is at least 4 feet deep should be considered a confined space entry, and all applicable requirements should be imposed and detailed in the HASP. See Section 4.4.2.3 for a brief discussion of confined space entry requirements.

Note: Excavation of contaminated soil can lead to the vaporization of toxic chemicals or the generation of contaminated dust, posing an inhalation hazard to sampling personnel. The SAP and HASP should address these potential hazards.

- **Environmental**
    - If soil contamination is suspected, large quantities of soil may have to be put into containers such as drums and stored or disposed of as hazardous waste
    - If the pit is refilled with excavated soil, contaminated surface material may be buried to a greater depth, or contaminated soil from subsurface layers may be exposed on the surface.

4.6.3. Subsurface Geological/Geotechnical Characterization

There are several drilling techniques that can be utilized to characterize the subsurface geology of a site. These drilling techniques do not provide undisturbed soil samples and should not be used in place of sonic drilling or cable tool methods.

4.6.3.1. Direct Mud Rotary

The direct mud rotary technique provides a flexible and rapid drilling method for a wide range of borehole diameters in consolidated and unconsolidated materials. Direct mud rotary drilling uses a rotating drill pipe with a hard-tooled drill bit attached at the bottom. Fluid is forced down through the drill pipe and then back up the borehole. The fluid is then discharged at the surface through a pipe or ditch into a sedimentation tank, pond, or pit. As the cuttings settle in the pond, the fluid overflows into a suction pit, where a pump recirculates the fluid back through the drill rods. The drilling fluid serves to accomplish the following:

- Cool and lubricate the bit
- Stabilize the borehole wall
- Prevent the inflow of formation fluids, thus minimizing cross-contamination of aquifers.

Casing is not required during drilling. When unconsolidated materials overlie a bedrock aquifer, mud rotary can be used to drill the bedrock. The hole can then be cased and a less intrusive drilling method, such as air rotary, can be used to complete the borehole. Reverse-circulation rotary drilling is a variant of the mud rotary method in which drilling fluid flows from the mud pit down the borehole outside of the drill rods and passes upward through the bit. Cuttings are carried into the drill rods and discharged back into the mud pit. Reverse-circulation rotary drilling equipment is similar to direct mud rotary drilling equipment except that most pieces are larger.

4.6.3.2. Directional Drilling

Directional drilling technology has the potential to offer borehole access to subsurface areas beneath buildings, tanks, landfills, and impoundments, where vertical drill rigs cannot reach. Directional drilling involves the use of equipment located at the surface to drill slanted or horizontal holes into the subsurface. Test applications have focused primarily on remedial activities, but the potential exists for use during characterization and monitoring activities. All directional drilling systems require the following:

- A steerable drill stem
- The capability to detect the location of the drill head or trajectory of the borehole.

Directional drilling equipment ranges in size from scaled-down rigs developed for the oil industry to relatively compact, simple equipment used to install utilities.

4.6.3.3. Direct Air Rotary with Rotary Bit/Down-Hole Hammer

Direct air rotary drilling should be considered for all situations involving consolidated rock. Air rotary drilling relies on a sharp bit to drill through earth and rock layers. A system of cables, engines, support mechanisms, lubricating devices, and pulleys control the rotation of the bit below the surface, keep the bit lubricated, and bring debris out of the borehole. The basic rig setup for air rotary with a tri-cone or roller-cone bit is similar to the setup for direct mud rotary drilling, except the circulation medium is air instead of water or drilling mud. Compressed air circulates down through the drill rods to cool the bit and then carries cuttings back up the borehole (a minimum 6-inch diameter hole required) to the surface. A cyclone separator is used to slow the air velocity and allow the cuttings to fall into a container. Several different bits might be used on a single borehole when drilling deep through different layers of rock. A larger diameter bit is generally used at the start of the drilling process with progressively smaller bits used to finish the hole. Use of a down-hole hammer in place of a roller-cone bit provides better penetration in hard geologic formations.

4.6.3.4. Rotary Diamond Drilling

Rotary diamond drilling is used for borehole drilling and coring in consolidated rock. This method uses a rotating bit consisting of a tube that is 10 – 26 feet long, with a diamond-studded ring fitted to the end of the core barrel. Water is circulated through the bit to cool the cutting surface. The diamond bit cuts through rock, with a solid core remaining in the tube.

4.6.4. Use of Soil Sampling Equipment

Soil sampling equipment should be selected according to the objectives of the sampling program. It is important to determine the depth at which samples will be collected prior to the start of a sampling program because sample depth will determine the types of equipment required. All sampling equipment should be decontaminated prior to use at each sampling location. Decontamination should be conducted in a controlled area before entering the site. Equipment should be decontaminated in the field between sampling locations as described in Section 4.9.

Note: Sampling equipment suppliers listed in EPA's manual *Description and Sampling of Contaminated Soils - A Field Pocket Guide* can be contacted to obtain pictures and information on soil sampling equipment.

4.6.4.1. Trowels, Scoops, and Spoons

Small hand tools can be used to collect surface samples and samples of undisturbed soil from the sides and bottoms of sampling pits. These small tools also are used to mix and handle soil obtained from deeper locations.

A trowel is a small shovel. A laboratory scoop is similar to a trowel, but the blade is usually more curved and has a closed upper end to contain the soil. Scoops come in different sizes and makes. Many are chrome plated, but these are unacceptable, since the plating can peel off and contaminate the soil sample. Stainless steel scoops are preferred. However, scoops made from other materials may be acceptable in certain instances. Stainless steel trowels and scoops can be purchased from scientific or forestry equipment supply houses. Stainless steel spoons can be purchased from housewares departments.

Hand tools should be decontaminated and wrapped in aluminum foil prior to use. If the equipment is not pre-wrapped in aluminum foil or another clean wrap material, sampling personnel should assume that it is not clean and should not use it.

4.6.4.2. Tube Samplers

Tube samplers are hollow tubes that are driven or screwed into the soil. The soil fills the tube and is retained when the sampler is withdrawn. Various types of tube samplers are available. The applications and limitations of the various types are summarized in Table 4-2. The split-spoon sampler and Veihmeyer sampler are discussed in Sections 4.6.4.3 and 4.6.4.4.

4.6.4.3. Split-Spoon Sampler

A split-spoon sampler is a length of carbon or stainless steel tubing split along its length and equipped with a drive shoe and drive head. Split-spoon samplers are available in a variety of lengths and diameters and generally are used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split-spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. If an auger is used, the split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted. A standard 2-foot split spoon is advanced ahead of the auger with a 140-pound hammer. Due to its weight, the split-spoon sampler is generally used with a drill rig. The advantages of split-spoon samplers include the ability to be driven into hard soils and the ease of extraction of the soil sample. When split-spoon sampling is performed to gain geologic information, all work should be performed in accordance with *ASTM D1586, Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils.*

Split-spoon samplers are available typically in diameters of 2 – 3½ inches. A larger barrel may be necessary to obtain the required sample volume.

Split-Spoon Sampler Use Procedures:

1. Use an auger or drill rig to open a borehole down to the depth to be sampled.
2. Assemble the split-spoon sampler by aligning both sides of the barrel, and then screw the drive shoe with retainer onto the bottom and the heavier head piece onto the top.
3. Place the sampler in a perpendicular position to the sample material.
4. Using a well ring, drive the tube. Do not drive past the bottom of the head piece, or compression of the sample will result.

5. Record the length of the tube used to penetrate the material being sampled and the number of blows required to obtain this depth.
6. Withdraw the sampler, and open it by unscrewing the bit and head and splitting the barrel. The amount of recovery and the soil type should be recorded.

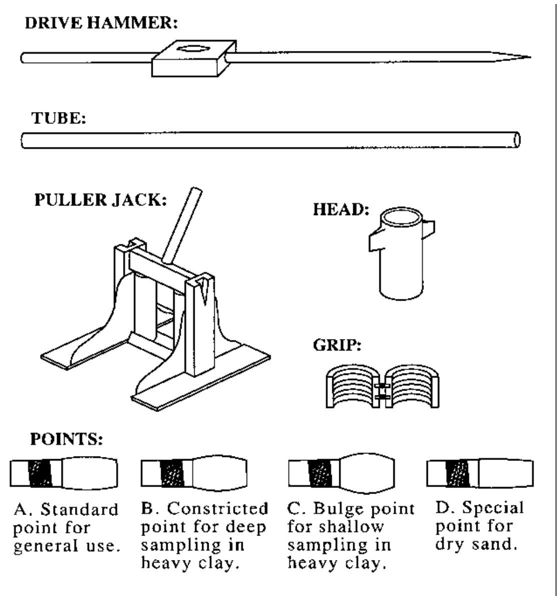
Table 4-2. Types of Tube Samplers

Sampling Device	Applications	Limitations
Split-spoon Sampler	Disturbed samples from cohesive soil	Ineffective in cohesionless sands; not suitable for collection of samples for laboratory tests requiring undisturbed soil
Soil Probe	Cohesive, soft soils or residue; representative sample in soft to medium cohesive soils and silts	Sampling depth generally limited to less than one meter
Shelby Tubes	Cohesive, soft soils or residue; special tips for wet or dry soils are available	Similar to Veihmeyer tube
Soil Recovery Probe	Similar to thin-walled tube; cores are collected in reusable liners, minimizing contact with the air	Similar to Veihmeyer tube
Veihmeyer	Cohesive soils or residue to depth of 3 meters	Difficult to drive into dense or hard material; will not retain dry, loose or granular material; may be difficult to pull from the ground
Peat Sampler	Wet, fibrous organic soils	

4.6.4.4. Veihmeyer Sampler

The Veihmeyer sampler was developed by Professor F. J. Veihmeyer of the University of California at Davis. The Veihmeyer sampler is recommended for core sampling of most types of soil; however, it may not be applicable to sampling stony, rocky, or very wet soil. A basic Veihmeyer sampler is shown in Figure 4-4 and contains the following components.

Figure 4-4. Veihmeyer Sampler



- Tube, 1.5 meters (5 feet)
- Tube, 3 meters (10 feet)
- Drive head
- Tip
- Drop hammer, 6.8 kilograms (15 pounds)
- Puller jack and grip, recommended for deep soil sampling.

The Veihmeyer sampler can achieve substantial depths with appropriate lengths of tubing, and various heads are available for different soil types. However, the Veihmeyer sampler can be difficult to clean, and some sampler components are constructed of materials that are not approved for the collection of certain types of analytes.

Veihmeyer Sampler Use Procedures

1. Assemble the sampler by screwing the tip and the drive head onto the sampling tube.
2. Insert the tapered handle (drive guide) of the drive hammer through the drive head.

3. Place the sampler in a perpendicular position to the material to be sampled.
4. 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 top of the hammer's guide.
5. Record the length of the tube that penetrated the material being sampled and the number of blows required to obtain this depth.
6. 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.
7. Rotate the sampler at least two revolutions to shear off the sample at the bottom.
8. Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head, and rotate about 90 degrees.
9. Withdraw the sampler by pulling the handle (hammer) upwards. When the sampler cannot be withdrawn by hand, as in deep sampling, use the puller jack and grip.
10. 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.

4.6.4.5. Thin-Wall Open Tube Samplers

Thin-walled core samplers are most commonly used for collection of undisturbed core samples in cohesive soils, silt, and sand above the water table. Sample collection procedures are similar to those for split-spoon sampling except that the tube is pushed into the soil using the weight of the drill rig, rather than driven. Some common thin-wall open tube samplers are discussed below.

4.6.4.5.1. Shelby Tube

Shelby tube samplers are used to obtain undisturbed samples in cohesive soils, silt, and sand above the water table. A Shelby tube consists of a thin-walled tube with a tapered cutting head. The device allows the sampler to penetrate the soil and aids in retaining the sample in the tube after the tube is advanced (without excessive force) to the desired depth. A Shelby tube is used mainly for collecting geologic information but may be used to obtain samples for chemical analysis.

Shelby tube samplers are easily cleaned, and VOC samples can be sent to the laboratory within the tube, thus minimizing air exposure. However, Shelby tube samplers can be difficult to drive into hard soils and are not durable in rocky soils. Further, certain types of soils can be difficult to extract from the tube.

Shelby Tube Sampler Use Procedures

1. Use an auger or drill rig to open a borehole down to the depth to be sampled.
2. Place the sampler over the borehole in a perpendicular position.
3. 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.
4. Let the tube sit for a few minutes to allow soils to expand in the tube.
5. Before pulling out the tube, rotate it at least two revolutions to shear off the sample at the bottom.
6. If the sample is to be shipped for geologic analysis, the ends of the tube should be sealed with wax to preserve the moisture content. In such instances, the procedures and preparation for shipment should be in accordance with *ASTM D1586-83*.

4.6.4.5.2. Continuous Tube

Continuous tubes are similar to Shelby tubes, except that the longer barrel is designed to operate inside the column of a hollow-stem auger.

Continuous Tube Sampling Procedures

1. Use an auger or drill rig to open a borehole down to the depth to be sampled.

2. Remove the auger from the drill rods, and insert a pre-cleaned thin-wall tube sampler. Install the proper cutting tip (an optional step is to first replace the auger tip with a planer auger to clean out and flatten the bottom of the hole before using the tube sampler).
3. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods, because the vibrations may cause the boring walls to collapse.
4. Remove the tube sampler, and unscrew the drill rods.
5. Remove the cutting tip and the core from the device.
6. Discard the top of the core (approximately 1 inch) because it possibly represents material collected before penetration of the layer of concern. Place the remaining core into the sample container(s).
7. If another sample is to be collected from a greater depth in the same hole, reattach the auger bit to the drill and assembly, and follow steps 2 through 7, making sure to decontaminate the auger and tube sampler between samples.
8. Abandon the hole according to applicable state regulations. Generally, shallow holes can be backfilled with the removed soil material.

4.6.4.5.3. Thin-Wall Piston/Specialized Thin-Wall Tube

Thin-wall piston samplers usually are used when soil conditions are unfavorable for conventional thin-wall samplers. Piston samplers are similar to conventional thin-wall samplers except they are equipped with internal pistons to generate a vacuum within the sampler as it is withdrawn from the soil. Many specialized thin-wall piston samplers have been developed for collecting undisturbed samples of a wide variety of soil types, as exemplified below:

- **Internal Sleeve Piston.** Heaving sands (used with a hollow-stem auger)
- **Wireline Piston.** Cohesive soils and non-cohesive sands (used with a hollow-stem auger)
- **Fixed-Piston.** Cohesive soils, silts, and sand above or below the water table
- **Stationary Piston, Free Piston, and Open Drive.** Undisturbed samples in stiff cohesive soils. Representative samples in medium to soft cohesive soils, silts, and sands.

The various types of specialized thin-wall tube samplers (and the soil types they are designed to collect) are listed below:

- **Pitcher.** Undisturbed samples in hard cohesive soils and cemented sands. Representative samples in soft to medium cohesive soils, silts, and sands. Frequently ineffective on loose soils
- **Denison.** Undisturbed samples in hard cohesive soils, cemented sands, and soft rocks. Not suitable for undisturbed sampling of loose or soft cohesive soils
- **Vicksburg.** Similar to Shelby tube but able to sample denser, coarser material.

4.7. Soil Sampling Procedures

The procedures described here generally apply to any type of soil sampling. Any departures from the procedures contained in the SAP should be documented and justified.

4.7.1. Preparation

Thorough preparation is the key to a successful project. Before beginning any sampling program, personnel should complete the following:

- Review the SAP and HASP to identify special equipment and procedures
- Obtain the required equipment. A sample list of soil sampling equipment is provided at the end of this chapter
- Inspect the site to ensure that present conditions are the same as indicated in the

SAP and that all designated sample locations are accessible

- Prepare decontamination facilities for personnel and equipment as required by the HASP.

Refer to Chapter 3 for a complete discussion of sampling preparation requirements applicable to all types of sampling programs.

4.7.2. Sample Collection

Field sampling personnel should be familiar with all of the technical issues and documentation provided in the SAP. The following sampling procedures can be used when there are no special conditions present or special requirements specified by the SAP. Refer to Chapter 3 for a complete discussion of sample collection requirements applicable to all types of sampling programs.

4.7.2.1. Surface Samples

The simplest and most direct method for collecting surface soil samples is with a spade and scoop. The accuracy and representative nature of these samples depends on the care and precision demonstrated by the sampler. A flat, pointed trowel can be used to cut a block of soil 2 inches deep. Chrome plated tools, common with garden implements such as potting trowels, should be avoided.

Sampling Procedures

1. Clear the area to be sampled of any surface debris such as twigs, rocks, and litter. Cut grass down to the level of the soil and remove.
2. Define a sample area such that a 2-inch deep soil sample will provide enough material for all required sample containers.
3. Dig a trench at least 2 inches deep around the sample block using a clean spade.
4. Cut the sample loose from the ground using a pre-cleaned stainless steel trowel. Place the soil in a clean stainless steel bowl.
5. Remove all roots and other debris. Describe the amount and kind of material removed in the FLB/FN.
6. If instructed by the SAP, sieve the sample as described in Section 4.7.4.2.
7. Fill the sample container(s).

4.7.2.2. Subsurface Samples

Handheld augers and thin-walled tube samplers can be used separately or in combination to collect subsurface samples. Where rocky soils do not limit the use of tube samplers, a combination of augers to remove soil material to the depth of interest and tube samplers for actual sample collection allows the most precise control of sample collection. Depths of up to 2 meters can be readily sampled, and depths of up to 6 meters can be achieved where conditions are favorable. A drill rig should be used to gain access where deeper samples are required or where soil conditions preclude the use of augers. Tables 5-1 and 5-2 summarize the applications and limitations of different types of augers and tube samplers for sampling under different soil conditions. Section 4.6.2.1 describes procedures for using augers. Section 4.6.4.5 discusses the selection and use of various types of thin-walled tube samplers.

4.7.2.3. VOCs

EnCore™ samplers can be used to collect soil samples for VOC testing. Separate procedures are provided below for collecting samples with high and low concentrations of VOCs.

Note: The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

When sampling for VOCs, soil samples should be taken from auger cuttings only if soil conditions make collection of undisturbed cores impossible. New techniques, such as direct-push technologies with the ability to continuously measure vadose zone VOC concentrations in the soil gas, are useful for defining the boring depth where discrete soil samples should be collected for laboratory analysis. Soil recovery probes with dedicated or reusable liners (see Table 4-2) will minimize contact of the sample with the atmosphere.

Sampling Procedures

Low-Concentration VOC Soil Samples

Steps 1 – 5 are performed prior to transporting VOC vials to the field for sample collection.

1. Add approximately 1 gram of sodium bisulfate to each vial. If samples that are markedly smaller or larger than 5 grams are to be collected, adjust the amount of preservative to correspond to approximately 0.2 grams of preservative for each gram of sample. Enough sodium bisulfate should be present to ensure a sample pH of 2.
2. Add 5 milliliters of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the VOAs.
3. Seal the vial with the screw cap and septum seal. If double-ended, fritted vials are used, seal both ends as recommended by the manufacturer.
4. Affix a label to each vial to eliminate the need to label the vials in the field. This will ensure that the tare weight of the vial includes the label. The weight of markings added to the label in the field is negligible.
5. Weigh the prepared vial to the nearest 0.01 gram, record the tare weight, and write it on the label.
6. Using an EnCore™ Sampler, collect approximately 5 grams of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere-generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.
7. Add about 5 grams (2 – 3 centimeters) of soil from the EnCore™ Sampler to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads, and immediately seal the vial with the septum and screw cap. Store samples on ice at ≤ 6°C.

Note: When samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low-concentration samples in vials that do not contain the preservative solution.

8. When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 grams of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers. Record the weight of the sealed vial containing the sample to the nearest 0.01 gram.

High-Concentration VOC Soil Samples

1. Add 10 milliliters of methanol to each vial.
2. Seal the vial with the screw cap and septum seal.
3. Affix a label to each vial to eliminate the need to label the vials in the field. This will ensure that the tare weight of the vial includes the label. The weight of markings added to the label in the field is negligible.
4. Weigh the prepared vial to the nearest 0.01 gram, record the tare weight, and write it on the label.

Note: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of > 0.01 gram) should not be used for sample collection.

5. Using an EnCore™ Sampler, collect approximately 5 grams of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere-generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6. Add about 5 grams (2 – 3 centimeters) of soil from the EnCore™ Sampler to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads, and immediately seal the vial with the septum and screw-cap. Store samples on ice at ≤ 6°C.
7. When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 grams of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers. Record the weight of the sealed vial containing the sample to the nearest 0.01 gram.

4.7.2.4. Other Parameters

Collect approximately one liter of soil for each sample, and follow procedures for sampling handling and preparation (see Section 4.7.4). Record the depth from the surface to the top and the bottom of the core, soil color, type, and other attributes in the FLB/FN. If the sample consists of two different types of soil (e.g., sand and clay, or different color layers), split the core as required so that only one soil type is present in the sample; record the depth of the change of soil types in the FLB/FN.

Following sample preparation and handling, cap sample bottles tightly, affix labels, and place the bottles in resealable plastic bags. The chemical preservation of soils generally is not required. However, samples should be cooled to ≤ 6°C as soon as possible.

4.7.3. Composite Sampling

Compositing is the process of physically combining and homogenizing several individual soil aliquots. Compositing provides for an average concentration of contaminants over a certain number of sampling points, which reduces both the number of required laboratory analyses and the sample variability. Compositing always should be implemented with caution. Composite samples should be collected only after full justification and documentation of rationale in the sampling plan. Composite sample collection techniques should not be used if justified solely on the basis of reducing sample testing costs. The method of compositing the sample should be documented and uniform to ensure future data comparability.

4.7.3.1. Composite Sampling Techniques

If composite samples are collected, the first step is to collect a soil sample from the sampling location specified in the sampling plan using the documented procedure. Any variation from the stated procedure or SAP must be recorded. Composite samples may be collected on the basis of biased sampling or a variety of statistical sampling techniques. Biased sampling is used when visual, odor, or volatile organic detection is found. A portion of the material is collected and combined with other positive areas. Two statistical sampling techniques that can be used for composite sampling are described below.

4.7.3.1.1. Random Sampling

Random sampling is the arbitrary collection of samples within defined boundaries of the area of concern. Random sample locations should be chosen with a random selection procedure (e.g., using a random number table). Each sampling point must be selected independently, so that all locations within the area of concern have an equal chance of being selected. Randomization is necessary to support probability or confidence statements about the sampling results. The key to interpreting these probability statements is the assumption that the site is homogeneous with respect to the parameters being monitored. The random sampling technique will not adequately characterize the true conditions of a heterogeneous site.

The following example illustrates how random soil sampling has been used to verify the achievement of soil remediation objectives:

Random Sampling Example. Approximately 300 tons of diesel contaminated soils were excavated from a railroad fueling operation and thinly spread over approximately one half acre to allow for biodegradation. A single composite soil sample was to be collected quarterly for total petroleum hydrocarbons to monitor remedial progress. The composite sample was to be prepared from 6 discrete soil samples. To collect the 6 soil samples from random locations across the half-acre area, a grid consisting of 40 equally sized squares was placed on a site map showing the area. Each square was numbered from 1 – 40, and a random number generator was used to pick locations from the grid. The sampling team then collected discrete soil samples from each of the six locations and prepared a single composite sample for laboratory analysis.

4.7.3.1.2. Stratified Random Sampling

Stratified random sampling often relies on historical information and prior analytical results (or screening data) to divide the sampling area into smaller areas called strata. Each stratum is more homogeneous than the site as a whole. Strata can be defined based on various factors, including sampling depth, contaminant concentration levels, and contaminant source areas. Stratified random sampling imparts some control upon the sampling scheme but still allows for random sampling within each stratum. Different sampling approaches may be selected to address the different strata at the site. Stratified random sampling is a useful and flexible design for estimating the pollutant concentration within each depth interval or area of concern.

4.7.3.2. Sample Compositing Procedures

In most cases of sample compositing, equal amounts of material are collected and mixed in the sample container. Protocols for combining the sample are based on site conditions, the parameters being measured, the regulatory limit, and DQOs. The rationale for the use of glass, stainless steel, or polyethylene equipment for combining samples, and the rationale for combining the soil in equal amounts, proportional amounts, or other amounts should be documented.

Specify the method for selecting the composite aliquots and the compositing factor in the sampling plan. The compositing factor is the number of aliquots to be composited into one sample (e.g., 3 – 1, 10 – 1). Determine this factor by evaluating the detection limits for parameters of interest and comparing them with the selected action level for that parameter. Compositing also requires that each discrete aliquot be the same in terms of volume or weight and that the aliquots be thoroughly homogenized. Because compositing dilutes high-concentration aliquots, the applicable detection limits should be reduced accordingly. If the composite value is to be compared to a selected action level, then the action level should be divided by the number of aliquots that make up the composite to determine the appropriate detection limit (e.g., if the action level for a particular substance is 50 parts per billion (ppb), an action level of 10 ppb should be used when analyzing a 5-aliquot composite). The detection level need not be reduced if the composite area is assumed to be homogeneous in concentration (e.g., stack emission plume deposits of particulate contamination across an area, or roadside spraying of waste oils).

In an example situation of a soil sampling program designed to detect petroleum products in an area of homogenous concentration, an appropriate sample compositing technique would be to use an adequately sized resealable freezer storage bag. Sampling personnel would collect a minimum of three equally sized samples (10 is preferred) from designated sample locations and place those samples in the sample bag. Personnel would then close and secure the bag using care to eliminate trapped air and combine the samples by tumbling and kneading until fully mixed.

Soil samples should be homogenized after compositing is complete to ensure soil uniformity.

Note: Do not composite or mix soil samples that are to be tested for volatile organics.

4.7.4. Sample Handling and Preparation

Proper sample handling and preparation ensures that each sample container is filled with soil that represents the entire sample and that large rock fragments are either removed or included in each container in an amount proportional to their presence in the soil, as specified by the SAP.

Techniques for sample handling and preparation are provided in the subsections below.

4.7.4.1. Soil Homogenization

Homogenization is the mixing or blending of a soil sample to provide uniform distribution of contaminants. Ideally, proper homogenization ensures that portions of the containerized samples are equal or identical in composition and representative of the total soil sample collected. Incomplete homogenization will increase sampling error. Samples to be composited or split should be homogenized after all aliquots have been combined. Manually homogenize samples using a stainless steel spoon or scoop and a stainless steel bucket, or use a disposable scoop and pan.

Note: Do not homogenize soil samples that are to be tested for volatile organics.

4.7.4.2. Sieving

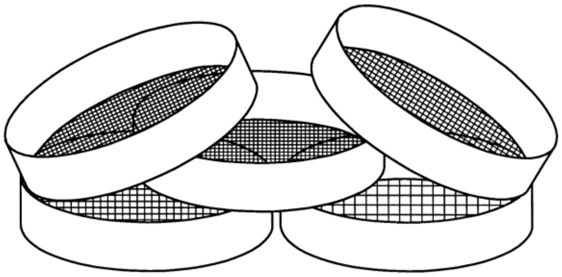
Sieving eliminates large rock fragments. Under some EPA protocols, only fine soil material should be analyzed; see Figure 4-5 for an illustration of sieves.

Note: Do not sieve soil samples that are to be tested for volatile organics.

Instructions for sieving are as follows:

1. Break up soil aggregates and pull apart any vegetation and root mat, if present. Weigh the non-soil vegetation fraction, and archive or discard, as required by the SAP.
2. Remove and weigh large rocks. Archive for possible analysis.

Figure 4-5. Sieves



3. Crush the entire soil sample with a rolling pin, stainless steel spoon, or similar tool. Mix thoroughly with a stainless steel spoon.
4. For samples of surface soil, sieve through a clean 0-mesh (No. 1, 2 mm) screen. Use a 3/8-inch standard sieve (9.5 mm) to screen samples that will be tested to determine whether the soil should be classified as hazardous waste under RCRA. Use a disposable stainless steel screen for samples to be analyzed for semivolatile organic contamination. Use Teflon® screens for samples to be analyzed for metal contamination.
5. If the soil is too wet or too cohesive to pass through the sieve, note that the required sieving was not done in the FLB/FN, and enter this notation on the Field Sampling Form. Inform the Sampling Program Manager that the sample could not be sieved.
6. Spread out the screened sample, mark off quarters, and take samples from each quarter in a consecutive manner until appropriate sample volume is collected. If specified, archive the remaining sample for future analysis.
7. Before shipping the samples to the laboratory, shake each sample container to mix thoroughly.

4.7.4.3. Splitting Samples

Many times samples are split between representatives of the property owner or between state or federal enforcement agencies so results may be independently verified. Soil samples split between laboratories are typically performed to evaluate laboratory quality. Soil samples should be split from a single sampling event following homogenization and sieving. Specific procedures for splitting samples for VOC, semivolatile organic, and metal analyses are described below.

- **Splitting Samples for VOC Analysis.** To split samples for VOC analysis, collect two core samples from adjacent locations. Alternatively, split a soil core longitudinally with a clean knife and place half of the core into each sample container, minimizing air contact as much as possible. The method of splitting the sample may greatly affect the results. Documentation and execution of the splitting technique should be uniform to ensure data comparability.

- **Splitting Samples for Semivolatile Organic Chemical (SVOC) and Metal Analyses.** To split samples for semivolatile and metal analyses, place the soil in a clean stainless steel bowl. Crush and thoroughly mix the soil with a clean stainless steel spoon. Document the method used for sample mixing and placing into the sample containers to ensure data comparability. One method is to form mixed soil into a cone and divide in half vertically. Each set of sample containers is then filled with the soil from half of the pile.

4.8. Restoration of Sample Locations

Sample locations should be restored to eliminate tripping hazards and remove conduits for contaminated runoff to reach subsurface soils. Take precautions to prevent introducing contaminated soil into uncontaminated soil horizons or leaving contaminated soil exposed on an otherwise clean surface.

4.8.1. Backfill Sample Holes

Soil pits and boreholes should be backfilled after samples have been collected. For shallow pits and boreholes where only one soil horizon is involved, the Sampling Program Manager may deem it appropriate to backfill with material from the pit or hole if this action has been specified in the SAP.

If backfilling with removed material is not authorized by the SAP, or where more than one soil horizon is involved, the pit or borehole should be backfilled with clean soil or sand. The fill material should be added in layers no more than 6 inches thick. Each layer should be compacted before more soil is added. This procedure will minimize subsequent settling.

4.8.2. Grouting Boreholes

Boreholes that penetrate a confining layer (e.g., silt, clay, or rock that acts as the base of an aquifer) should be grouted to prevent migration of contaminated water between aquifers. Grouting consists of filling the borehole with concrete or bentonite slurry as specified by the Sampling Program Manager and the SAP. Record grouting details (e.g., depth of borehole, depth to confining layer, mix ratio, and amount of the grout used) in the FLB/FN for each borehole.

4.8.3. Replace Sod

Sod should be replaced in grass areas after the boreholes and pits are filled. The sod originally removed from the area should be used if it is still alive. Otherwise, commercial sod can be used or the area should be seeded, depending on the requirements of the facility.

4.8.4. Replace Pavement

Areas of pavement that were removed to obtain access to soil should be repaired after the pit or borehole is filled. As required by the facility, the patch may be cold patch asphalt, hot asphalt, or concrete.

4.9. Decontamination

It is generally not possible to have a separate set of precleaned stainless steel sampling equipment for each sample if significant numbers of samples are to be collected. Sampling equipment should be decontaminated prior to use to prevent contaminants from being transferred from one sample to the next. If sampling equipment must be reused, it must be decontaminated prior to sampling and between each sample location.

All field decontamination should take place on a decontamination pad lined with plastic. Solvent rinse liquids must be collected in dedicated containers. All aqueous decontamination liquids should be stored in DOT approved compatible containers (e.g., 55-gallon drum or appropriate size). If nitric acid is used to rinse the sampling equipment, the rinse liquids should be neutralized by adding lime to each drum (1 cup of slaked lime, $Ca(OH)_2$, per drum). If any hazardous chemical is used on site, the SAP must detail proper waste material handling procedures.

4.9.1. Decontamination Solutions and Supplies

Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment. Most equipment and supplies can be easily procured such as softwash and rinse solutions. Children's wading pools also can be used. Large plastic garbage cans or other similar containers lined with plastic bags can help segregate contaminated equipment. Contaminated liquid can be stored temporarily in metal or plastic cans or drums. The following standard materials and equipment are recommended for decontamination activities:

- Solutions
  - Non-phosphate detergent
  - Selected solvents such as acetone, hexane, nitric acid, and isopropyl alcohol
  - Dilute bases
  - Tap water
  - Distilled or deionized water.
- Tools and Supplies
  - Long and short handled brushes
  - Bottle brushes
  - Drop cloth or plastic sheeting
  - Paper towels
  - Plastic or galvanized tubs or buckets
  - Pressurized water sprayers
  - Solvent sprayers
  - Aluminum foil
  - Trash bags
  - Trash containers
  - 55-gallon drums
  - Metal or plastic buckets or containers for storage and disposal of decontamination solutions.
- Solvent Rinses
  - 10% nitric acid is typically used for inorganic compounds such as metals. An acid rinse may not be required if inorganics are not a contaminant of concern.
  - Acetone (pesticide grade), hexane (pesticide grade), and methanol, only if sample is to be analyzed for organics.

Note: All cleaners or solvents must be approved by the Program Manager or designee. At least once a day, collect samples of the cleaner and water that have been used as a final rinse for the equipment for submission to the laboratory as an equipment rinsate blank.

Table 4-3 provides a summary of the solvent types and recommended solvent rinses for various types of pollutants.

Table 4-3. Soluble Contaminants and Recommended Solvent Rinse

Solvent[1]	Examples of Solvents	Soluble Contaminants
Water	Deionized water, tap water.	Low-chain hydrocarbons, inorganic compounds, salts, some organic acids, and other polar compounds.
Dilute Acids	Nitric acid Acetic acid, boric acid.	Basic (caustic) compounds (e.g., amines and hydrazines).
Dilute Bases	Sodium bicarbonate (e.g., soap detergent).	Acidic compounds, phenol, thiols, some nitro and sulfonic compounds.
Organic Solvents[2]	Alcohols, ethers ketones, aromatics, straight-chain alkalines (e.g., hexane), common petroleum products (e.g., fuel, oil, kerosene).	Nonpolar compounds (e.g., some organic compounds).
Organic Solvents[2]	Hexane	PCBs

[1] Material Safety Data Sheets (MSDSs) are required for all decontamination solvents or solutions as required by the Hazard Communication Standard.

[2] **WARNING:** Some organic solvents can permeate or degrade protective clothing.

4.9.2. Procedure

The following procedures should be used to clean field sampling equipment between samples. Note that different procedures are used depending on whether the equipment will be used to collect samples for metals or organic analysis and the level of detection required for the compliance program. Cleaning and decontamination procedures for equipment used to collect soil samples for analysis for organic and inorganic chemicals are as follows:

- **Segregate Equipment Drop**
    - Place plastic sheeting on the ground. Size will depend on amount of equipment to be decontaminated.
    - Provide containers lined with plastic if equipment is to be segregated. Segregation may be required if sensitive equipment or mildly contaminated equipment is used at the same time as equipment that is likely to be heavily contaminated.
    - Deposit equipment used on site (e.g., tools, sampling devices, and containers, monitoring instruments radios, etc.) on the plastic drop cloth or sheet, or in different containers with plastic liners. Each will be contaminated to a different degree; segregation at the drop reduces the probability of cross contamination.
    - Loose leaf sampling data sheets or maps can be placed in plastic zip lock bags if contamination is evident.
- **Physical Removal With a High-Pressure Washer (Optional)**
    - A high-pressure wash may be required for compounds that are difficult to remove by washing with brushes. Hot water from the high-pressure washers is excellent at removing greasy or oily compounds.
    - High-pressure washers require water and electricity
    - A decontamination pad may be required for the high-pressure wash area. An example of a wash pad may consist of an approximately 1½-foot-deep basin lined with plastic sheeting and sloped to a sump at one corner. A layer of sand can be placed over the plastic, and the basin is filled with gravel or shell.
    - The sump is also lined with plastic, and a barrel is placed in the hole to prevent collapse. A sump pump is used to remove the water from the sump for transfer into a drum.
    - Heavy machinery is usually decontaminated at the end of the day unless site sampling requires the machinery be decontaminated more frequently. A separate decontamination pad may be required for heavy equipment.
    - Use high-pressure wash on grossly contaminated equipment. Do not use it on sensitive or non-waterproof equipment.
- **Physical Removal with Brushes and a Wash Basin**
    - Fill a wash basin, a large bucket, or child's swimming pool with non-phosphate detergent and tap water. Approximately 10 – 50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.
    - Scrub equipment with soap and water using bottle and bristle brushes
    - Only sensitive equipment (e.g., radios, air monitoring and sampling equipment) which is waterproof should be washed
    - Equipment that is not waterproof should have plastic bags removed and wiped down with a damp cloth
    - Acids and organic rinses may also ruin sensitive equipment. Consult the manufacturers for recommended solutions.

- **Equipment Rinse**
    - Fill a wash basin, a large bucket, or child's swimming pool with tap water. Approximately 10 – 50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.
    - Rinse soap off equipment with water by immersing the equipment while brushing. Repeat as many times as necessary.
- **Low-Pressure Rinse**
    - Fill a low-pressure sprayer with distilled or deionized water during the rinsing process. Approximately 10 – 20 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.
    - Rinse sampling equipment with distilled or deionized water with a low-pressure sprayer.

Note: Personal protective equipment (PPE) recommended for handling nitric acid includes nitrile gloves, boot covers, rubber apron or coated Tyvek® coveralls, and eye protection (e.g., face shields or goggles).

- **Nitric Acid Sprayers (Required Only if Metals Are a Contaminant of Concern)**
    - For low-level contamination or where equipment rinsate blanks demonstrate metals contamination, rinse stainless steel equipment with 10% nitric acid (trace metal or higher grade HNO_3 diluted with distilled or deionized water)
    - Fill a spray bottle with 10% nitric acid. An acid rinse may not be required if inorganics are not a contaminant of concern. The amount of acid will depend on the amount of equipment to be decontaminated.
    - Provide a 5-gallon bucket or basin to collect acid during the rinsing process
    - Rinse sampling equipment with nitric acid. Begin spraying (inside and outside) at one end of the equipment allowing the acid to drip to the other end into a 5-gallon bucket.
- **Low-Pressure Sprayers**
    - Fill a low-pressure sprayer with distilled or deionized water during the rinsate process.
    - Rinse sampling equipment with distilled or deionized water with a low-pressure sprayer.
- **Organic Solvent Sprayers**
    - Fill a spray bottle with an organic solvent. After each solvent rinse, the equipment should be rinsed with distilled or deionized water and air dried. Amount of solvent will depend on the amount of equipment to de-decontaminate.
    - Provide a 5-gallon bucket or basin to collect the solvent during the rinsing process
    - Solvent rinses may not be required unless organics are a contaminant of concern
    - Rinse sampling equipment with a solvent. Begin spraying (inside and outside) at one end of the equipment allowing the solvent to drip to the other end into a 5-gallon bucket.
    - Allow the solvent to evaporate from the equipment before going to the next step. A QC rinsate sample may be required.
- **Low-Pressure Sprayers**
    - Fill a low-pressure sprayer with distilled or deionized water. Use a 5-gallon container to collect water during the rinsate process.
    - Rinse sampling equipment with distilled or deionized water with a low-pressure washer.
- **Clean Equipment Drop**
    - Lay a clean piece of plastic sheeting over the bottom plastic layer. This will allow easy removal of the plastic in the event that it becomes dirty.

  - Provide aluminum foil, plastic, or other protective material to wrap clean equipment
  - Lay clean equipment on plastic sheeting. Let air dry, wrap sampling equipment with aluminum foil, plastic, or other protective material.
- **Record Information**
  - Information concerning decontamination methodology, date, time, and personnel should be recorded in the FLB/FN.

4.9.3. Preparation of Equipment Rinsate Blanks

Equipment rinsate blanks provide a check on the cleanliness of sampling equipment and the purity of the water or solvent used for the final rinse(s). See Chapter 3 for information on collecting equipment rinsate blanks.

At least one equipment rinsate blank should be submitted each day for each type of rinse solvent used (e.g., water, acetone). The SAP should specify the size and material of each sample bottle required for equipment rinsate blanks and any required preservatives for these liquid samples.

Equipment rinsate blanks consisting of solvents such as acetone or hexane will be classified as flammable liquids and will require special packaging (in metal cans). Special labeling and manifesting requirements apply for shipments of flammable liquids sent to the lab by common carrier.

4.10. Waste Material Storage and Disposal

All material generated during soil sampling projects should be stored as hazardous waste until disposal decisions are made based on generator knowledge or test results. This includes soil cuttings, equipment decontamination wastes, and used disposable PPE.

4.10.1. Soil Cuttings

Soil cuttings from boreholes and test pits may be classified as hazardous waste and should therefore be containerized and stored in accordance with EPA and facility requirements. Soil cuttings must not be used to fill the boreholes or test pits unless specifically authorized by the SAP or regulatory authority.

Soil cuttings should be placed on plastic sheets when generated. The soil should be stored in metal drums that are marked to indicate the source of the contents. The plastic sheets should be disposed with the used PPE.

4.10.2. Used Disposable PPE

All disposable solid contaminated equipment (e.g., plastic sheets, screens, coveralls, boot covers, etc.) should be placed in plastic bags temporarily, then sealed in metal barrels for final storage, transport, and disposal based on generator knowledge or test results.

4.11. Sampling Equipment List

Section 3.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional equipment applicable to soil sampling. In most cases, only a portion of the equipment listed below would be required for a given sampling event.

- Equipment for accessing soils
  - Backhoe
  - Augers
  - Drill rigs
- Soil sampling equipment
  - Trowels, scoops, and spoons
  - Split-spoon sampler and hammer to advance.
  - Veihmeyer soil sampler outfit
  - Shelby tube
  - Continuous tube
  - Thin-wall tube sample
  - EnCore™ sampler
- Additional equipment
  - Maps and plot plan
  - Survey equipment or GPS to locate sampling points
  - Survey stakes or flags

- Decontamination supplies and equipment
- Canvas or plastic sheet
- Shovel (to refill sampling holes)
- Stainless steel mixing bowl
- Detergent
- Acetone, hexane, or biodegradable organic cleaner
- Nitric acid
- Tap water
- Distilled or deionized water (ASTM Type II)
- Storage and disposal containers for wash water, acid, solvents, and soil cuttings
- Lime to neutralize nitric acid wastes,
- Grouting materials such as concrete or bentonite slurry
- Tape to measure sample depth
- Vermiculite.

4.12. References

1 ASTM D5314 Standard Guide for Soil Gas Monitoring in the Vadose Zone2 California Environmental Protection Agency, Department of Toxic Substances Control, "Advisory Active Soil Gas Investigation," April 2012, http://www.dtsc.ca.gov/SiteCleanup/upload/VI_ActiveSoilGasAdvisory_FINAL_043012.pdf

3 DoD Vapor Intrusion Handbook, http://www.clu-in.org/download/char/dodvihdbk200901.pdf

4 USACE Sample Collection and Preparation Strategies for Volatile Organic Compounds in Solids, October 1998

Chapter 5. Aquatic Sediment Sampling

5.1. Purpose

This chapter provides general guidelines and procedures for sampling approaches, methods, and equipment for aquatic sediment characterization to assess risks at DoD sites. This chapter provides general guidance, and references appropriate detailed sampling approaches, methods, and equipment. Topics include sampling method selection; sediment manipulation methods to prepare for chemical analysis and toxicity testing; sampling of sediment pore water; and sampling of benthic invertebrate infauna (organisms that burrow in sediment (e.g., clams, worms)) and epifauna (organisms living on sediment surface and freely roaming, (e.g., some insects and crabs)). Key references for additional information are provided at the end of each section.

5.2. Scope

Aquatic sediments consist of particles of mineral or organic material that accumulate under surface water. The particles, which include solids and sludges that are suspended or settled in the water, are derived from rocks or biological materials that are either transported by flowing water bodies or situated beneath a static aqueous layer. Sediment types are classified by particle size, mineralogy, source materials, and site-specific variables. Sediment and sediment-related sampling and analysis is used to address risk to human health or the environment. Specific uses of sediment data include the following:

- Characterizing extent of contamination or risk associated with site and ambient conditions
- Determining impacts to benthic communities
- Evaluating toxicity through bioassays
- Estimating bioaccumulation of certain chemicals
- Evaluating impacts from pore water
- Evaluating impacts from sediment disturbance (e.g., currents, dredging, prop-wash and scour).

Some sediment sampling procedures may need to be modified to accommodate site conditions and equipment or logistic limitations (e.g., depth to sediment, difficulty of penetration of sediment). Field sampling should be conducted in accordance with appropriate federal, state, or local government regulations.

Sediment samples may be collected from streams, lakes, ponds, creeks, lagoons, surface impoundments, and coastal (marine/estuarine) waters, near the surface, or at deeper intervals. Benthic invertebrate collections are typically done at the surface or at shallow depths where organisms reside, and result in benthic community measures that may be used to compare to a reference or ambient community in order to determine whether the site sediments are impacted. Bioassay and bioaccumulation methods may be used to expose representative aquatic organisms to sediment, usually in a laboratory setting, to determine their response in comparison to a reference or ambient sediment response. Sediment pore-water sampling is also an option to determine risk of contaminants, or to assess the movement of groundwater through sediment in a groundwater discharge zone or hyporheic zone of flow.

5.3. Project Planning for Sediment-Related Sampling

As discussed in Chapter 2, the systematic planning process is critical to acquiring representative samples. However, sediment sampling introduces additional concerns. For example, sediment typically has low-level contaminant action levels compared to waste and soil media, and related problems with introducing trace-level cross-contamination from the bulky mechanical gear required for sampling. Clear and concise objectives should be identified to define the intent of the sediment-sampling program. The overall process includes the following steps:

5.3.1. Identify Project Goals

A key step in identifying project goals is to specify the problem and indicate decisions that need to be made regarding it. A CSM is used to compile and summarize existing information and hypotheses regarding contaminant sources and migration pathways to prospectively-exposed human and environmental receptors. In many cases regarding sediments, the prospective impacts to these receptors is the decision. However, legal considerations may also be important (e.g., state laws specifying sediment cleanup requirements, or federal or state laws that establish hazardous or dangerous waste categories).

CSMs should consider the following as they influence the site decisions. Table 5-1 below shows an expanded list.

- Historical site use including known or suspected contamination in site media
- Site topographic and meteorological features, including water depth and sediment thickness
- The hydrodynamic conditions of the water body as related to physical sediment fate and transport, (e.g., runoff, current and deposition patterns, upwelling, and seepage patterns)
- Processes that influence the transport and concentrations of contaminants present (e.g., advection, dilution, sediment transport, biodegradation, and dispersion)
- The human and fish or wildlife utilization of the site (e.g., children accessing sediments on beaches, or wildlife consuming fish from the site)
- Processes that affect contaminant bioavailability.

5.3.2. Determine Data Needs

The project planning team should identify key inputs or data needs and provide narrative statements of how data will be used in decisions, i.e., data quality objectives. An important part of this process is the boundaries (spatial or temporal) of the study, which affect the scope of the investigation.

Typical decisions for sampling sediment include the following:

- Identifying contamination presence and extent
- Identifying sources of contamination
- Identifying whether contamination levels suggest adverse biological effect
- Identifying trends
- Establishing goals for sediment remediation
- Evaluating possible effects of dredged or fill material discharge.

For determination of adverse biological effects, there are several possible phases of inquiry and response, and the process selected should be described in the data quality objectives. What follows summarizes the process.

5.3.2.1. Develop a Screening-Level Chemical Evaluation

Develop a Screening-Level Chemical Evaluation that compares site sediment and/or fish/shellfish tissue concentrations to background and screening criteria in order to generate a list of contaminants of potential concern (COPC), and to determine whether additional investigations or actions are needed. Until recently, sediment screening has been based on bulk sediment concentrations compared to database-derived measures of effect (e.g., the National Oceanographic and Atmospheric Administration (NOAA) Screening Quick Reference Table (SQuIRT) values), or equilibrium-partitioning derived values that estimate the proportion of a substance in sediment pore water. Recently, researchers have developed better screening criteria that incorporate bioavailability as a predictor of toxicity and bioaccumulation. The National Research Council (NRC 2003) defines bioavailability processes as, “individual physical, chemical, and biological interactions that determine the exposure of plants and animals to chemicals associated with soils and sediments.” Traditional methods to assess the potential effect of sediment contamination on human or ecological health use total bulk sediment contaminant concentrations. However, abundant research has shown that only a fraction (the bioavailable fraction) of an environmental contaminant may be taken up and may subsequently result in an effect on an organism; and that this concentration is often much less than the bulk concentration of these contaminants in the sediment.

Depending upon the project decision and the complexity of the investigation, bioavailability measures may be desirable to include in the investigation. An example bioavailability screen for divalent inorganic compounds is to collect data on acid volatile sulfides and simultaneously extracted metals. An example for neutral organic compounds is to use a method for collection of sediment pore water. Table 5-1 summarizes a variety of measures that affect bioavailability, and related measures that may require inquiry.

5.3.2.2. Characterize Exposure and Effect

Characterize Exposure and Effect using the COPCs identified, and conducting human health or ecological risk assessment, and determining the risk or hazard associated with the sediment. This step may also include comparisons of benthic communities of invertebrates at the site to a reference area community as a measure of effect. In Table 5-1, the rows titled, “Evaluate Ecological and Human Health Pathways and Endpoints” apply to this step.

5.3.2.3. Assess Risk Management

Assess Risk Management options regarding the site; these may lead to no action, or to a sediment removal, or in the case of sites managed under CERCLA, as amended, to a Feasibility Study and Record of Decision (ROD) regarding the extent of a response action.

5.3.3. Develop Data Collection Options

Specific considerations that may influence data collection strategies for sediments may include:

- Hydrologic information (e.g., runoff, current and deposition patterns, upwelling, seepage patterns)
- Water depth
- Thickness and type of sediment, especially regarding sediment grain size, as it affects sampling efficiency and sample representativeness
- Anthropogenic and natural loading (proximity of outfalls, seasonal patterns, relevant previous monitoring information)
- Contaminant bioavailability
- Type of benthic community being represented, as it affects the size and number of samples required for characterization; note that habitat structure and function may influence the community composition
- Potential for release of sediment chemicals to surface water via diffusion or advection
- Fate of sediment (e.g., lithic structure, current regime with regard to depositional and erosional conditions, and related activities such as dredging).

Specific measurements may include:

- Chemical data on bulk sediment and pore water
- Sediment toxicity (organism response)
- Tissue bioaccumulation (tissue levels upon exposure to sediment)
- Bioavailability measures
- Benthic community analysis (e.g., habitat structure, and benthic surveys). Although this chapter is limited to sediment and associated invertebrate communities, there are benthic fish species whose life cycles are coupled to sediment that may also be useful bioindicators. Examples include mummichog, sheepshead minnow, and oyster toadfish.

Table 5-1 below illustrates a range of activities and measures potentially associated with developing project objectives and data needs. Note that some of these evaluations and investigation methodologies are beyond the scope of this chapter, but are potential additional lines of evidence.

Appendix B of US EPA’s *Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses* (Ref. 1) illustrates some typical data quality objectives and measurement quality objectives for sediment investigations.

Table 5-1. Approaches Used to Assess Bioavailability of Chemicals for Sediment.
(Adopted from ITRC 2011.) The added phrase "as appropriate to the site" is intended to indicate that not all sites require this evaluation.

Scoping Element	Source of Information	Activity	Bioavailability Related Analyses/ Considerations
Review Site History	Review Available Data (e.g., hazard ranking documents)	Establish site boundaries, identify adjacent facilities Identify initial COPC Identify geochemical parameters Understand reference (background) conditions	Nature and extent of COPCs and exposure COPC mobility forms and pathways Data gap analysis (organic carbon, acid volatile sulfides and simultaneously extracted metals, redox, salinity, pH)
Identify Site Transport Processes	Groundwater Data, *as appropriate to the site*	Understand direction and flow Understand hyporheic zone mixing, if present	Flux measures in sediments via piezometers, direct pore water, or passive samplers
	Sediment Data	Assess bulk sediment	Analyses of COPC, total organic carbon, acid volatile sulfides and simultaneously-extracted metals, metals speciation as appropriate, and grain size
		Assess pore water, *as appropriate to the site*	Sample and analyze COPC via piezometers, direct pore water measurement by extraction or by passive samplers Estimate pore water fraction by Equilibrium Partitioning, acid volatile sulfides and simultaneously-extracted metals, the biotic ligand model, or other appropriate model(s)
		Assess diffusive flux, *as appropriate to the site*	Analyses of dissolved COPC immediately above sediment/water interface, *as appropriate to the site* Estimate flux based on models, such as the USACE RECOVERY model, a contaminated sediment-Water interaction model
	Potential Sediment Hydraulics	Assess Flood or ice scour, *as appropriate to the site*	Estimates and/or analyses of COPC desorption from resuspended sediments
		Assess wave/current-induced resuspension, *as appropriate to the site*	Redeposition of contaminated sediment
		Assess boat propeller wash, *as appropriate to the site*	Measures of total and dissolved COPCs, TOC and DOC estimates and/or analyses of COPC desorption from resuspended sediments

Scoping Element	Source of Information	Activity	Bioavailability Related Analyses/ Considerations
		Assess resuspension flux, *as appropriate to the site*	Resuspension of contaminated sediment Exposure to buried contaminated sediments Non-equilibrium conditions for organics Oxidation of metals Direct measures of COPCs in overlying waste and newly exposed sediments Changes in oxidation conditions Changes in salinity; estuarine/tidal wedge Estimates and/or analyses of COPC desorption from resuspended sediments
Evaluate Ecological and Human Health Pathways and Endpoints	Ecological receptor-based information	Evaluate receptor and exposure pathways ▪ Benthic invertebrates ▪ Fish and water column invertebrates ▪ Wildlife ▪ Plants	Plant tissue residue analysis, plant bioassays Community analyses, bioassays, tissue residue analysis, Equilibrium Partitioning and Biota-Sediment Accumulation Factor uptake models Tissue residue analysis (COPC and lipid), Biota-Sediment Accumulation Factor (BSAF), kinetic uptake models Tissue residue analysis (COPC and lipid), oral dose models, laboratory bioassays, field assessment of fecundity Animal testing for bioavailability (e.g., swine); tissue residue measure (COPC and lipid), oral dose models, laboratory bioassays
	Human Health-based information	Evaluate receptor and exposure pathways: ▪ Direct contact ▪ Ingestion	Water analysis for COPCs, partitioning variables (e.g., Octanol-Water Partition Coefficient (Kow)), oral dose In-vitro/in-vivo testing for bioavailability (e.g., swine oral and dermal dose models); bulk sediment analysis, partitioning measures , Tissue residue(s), fraction of COPC available for uptake (bioaccessibility), Identification of ingestion rates

For more information on project planning for sediment-related sampling, consult the following references:

- Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual (Ref. 1)
- Guidance Manual to Support the Assessment of Contaminated Sediments in Freshwater Ecosystems Volume I - An Ecosystem-Based Framework for Assessing and Managing Contaminated Sediments (Ref. 2)
- Contaminated Sediment Remediation Guidance for Hazardous Waste Sites (Ref. 3)
- Implementation Guide for Assessing and Managing Contaminated Sediment at Navy Facilities (Ref. 4)
- Critical Issues for Contaminated Sediment Management (Ref. 5).

5.4. Developing a Sampling Strategy

This section describes development of data collection options, and finalizing the data collection program. Development of a sampling strategy for purposes of characterizing a sediment site should follow the fundamentals of the scientific approach, including appropriate quality control and quality assurance.

Factors such as physical site constraints, safety, and cost may influence sampling strategies. Project strategies should consider the current and potential future phases of an investigation, and select the methodologies appropriate to these phases. A "phased" investigative approach can be used to refine the CSM and address the data quality objectives for the investigation. It is important to consider exit strategies should the data indicate that further investigation is unnecessary. For example, comparison to conservative bulk sediment screening values or background comparisons are often used to determine the need for later stages of investigation. Should the screening values or background values be exceeded, intensified sampling could be initiated to identify or delimit a source area and release pattern, and to confirm whether the site concentrations represent site-related contamination.

Alternatively, a data collection program may be more comprehensive in the first phase, for example, when contamination is believed to be present, the source study and release pattern may occur at the beginning of the project.

Reference locations may be important for contaminant screening, or later in the process, for determining project-relatedness of sediment chemistry or risk. Selection of a sediment reference location requires matching sediment characteristics (e.g., grain-size ranges, TOC, pH, eH, and, where toxicity testing is desired, toxicity limits) from an unimpacted or non-site reference location to the site sediments. A reference site selection study should define what is intended: either a pristine reference ("natural" or "non-anthropogenically affected" reference) or a site that represents the conditions in the watershed apart from the site being investigated ("ambient" or "anthropogenically-affected" reference). In most DoD site investigations, the latter is desired; in some state programs, the former is mandated. To assure that grain-size, TOC, and eH conditions are comparable may require sampling several stations to achieve an acceptable match. Performance criteria for reference stations should be included in project plans. Sampling intensity at the reference station should also be sufficient to establish a statistically valid comparison between it and the investigation site.

Advanced methods to determine whether receptors are likely to show adverse effects, or to elucidate release patterns and refine the CSM with regard to contaminant mobility and bioavailability may also be included in a second or contingent phase of the investigation. Examples include bedded sediment bioaccumulation testing, collection of field organisms for tissue analysis, measures of pore water, or benthic mapping (section 5.8).

US EPA QA/G-5s (http://www.epa.gov/quality/qs-docs/g5s-final.pdf) provides useful guidance on statistical sample design including numbers and types of samples, selection of sample locations, sampling frequency, and sampling and analytical protocols. In addition, there are free software programs available such as ProUCL and Visual Sample Plan that permit estimation of numbers of samples required.

The analyte list for sediment and pore water sampling schema should anticipate the intended data uses. For instance, for toxicity testing, grain-size and total organic carbon are recommended; for some analytes (e.g., organotins), bulk sediment may not be an appropriate medium and pore-water analysis may be required. Some analytes (e.g., mercury) have a short holding time, which may affect the field schedule for delivery to analytical laboratories. There are also limitations on holding time for sediment prior to initiating biological testing. The sampling method and organism size to be retained from macro invertebrate surveys should anticipate the uses of data (e.g., taxonomic composition and mass, species diversity measures).

For more information on developing a sampling strategy, consult the following references:

- Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses (Ref. 1)
- Sampling for Contaminants In Sediments and Sediment Pore Water (Ref. 6)
- A Compendium of Chemical, Physical and Biological Methods for Assessing and Monitoring the Remediation of Contaminated Sediment Sites (Ref. 7)
- Contaminated Sediment Remediation Guidance for Hazardous Waste Site (Ref. 3)
- Ocean Testing Manual Evaluation of Dredged Material Proposed for Ocean Disposal (Ref. 8)
- Inland Testing Manual: Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S (Ref. 9)
- Guide for Incorporating Bioavailability Adjustments into Human Health and Ecological Risk Assessments at U.S. Navy and Marine Corps Facilities (Ref. 10)
- Incorporating Bioavailability Considerations into the Evaluation and Remediation of Contaminated Sediment Sites (Ref. 11)
- Assessment and Remediation of Contaminated Sediments (ARCS) Program - Assessment Guidance (Ref. 12).

5.5. Methods for Bulk Sediment Sampling

This section presents guidelines for collecting representative sediment samples, and targets the selection of appropriate equipment based upon a range of site conditions such as depth of water, depth of sediment to be sampled, and flow regimes.

Desirable attributes for sediment sampling gear include the following:

- Creates a minimal pressure wave when descending through the water
- Forms a nearly leak-proof seal when the sediment sample is taken
- Prevents winnowing and excessive sample disturbance when ascending
- Allows easy access to the sample surface so that undisturbed subsamples may be taken
- Allows vertical sectioning of undisturbed samples for profile examination
- Penetration well below the desired sampling depth is preferred to prevent sample disturbance as the device closes. A means of weight adjustment to assure penetration depths may be valuable.

5.5.1. Sediment Grab Sampling Methods

Grab samplers are excellent for providing quantitative data at a relatively low cost. Considerations for selection of core versus grab samplers are described below in Table 5-2.

Limitations of grab samplers include:

- Variability among penetration depth for samples depending on sediment properties
- Oblique angles of penetration which result in varying penetration depths within a sample
- Folding or disturbing the sample, resulting in the loss of information concerning the vertical structure of benthic communities in the sediments.

Some additional things to consider when choosing a sampler include:

- Water depth
  - In less than 4 m of water, with low current conditions and smooth water, the Birge-Ekman, petite Ponar, Ponar, Van Veen, and Peterson mini-Shipek grabs are recommended
  - For deeper waters or greater current, the Ponar or Van Veen are recommended.
- Sub-sampling from sampler directly
  - If sub-sampling from the sampler is desired, the Birge-Ekman, mini-Ponar, Ponar, Van Veen, or Shipek are recommended
  - If not, a Peterson, Smith-MacIntyre, or mini-Shipek are recommended.
- Target sample depths
  - Up to about 10 cm sample depth, a Birge-Ekman, Ponar, or Shipek or miniature versions of any of these are suitable
  - Up to about 30 cm, a Smith MacIntyre, Van Veen, or Petersen grabs are suitable.
- Target sample volumes to meet analytical requirements

- Up to about 3 L, the mini Birge-Ekman, petite Ponar, or mini-Shipek samplers are suitable
- 3 – 10 L samples may be collected with the Birge-Ekman, Ponar, or Petersen grabs
- Above 10 L, Smith-MacIntyre or Van Veen grabs are suitable.

The most common surface area sampled is about 0.1 m^2 in the Van Veen and Smith-MacIntyre grabs. Van Veen and box corers are also capable of sampling 0.06 m^2, compared to hand-held corers, which sample about 0.001 m^2.

Table 5-2. Selection Criteria for Core Sampler or Grab Sampler

Use a Core Sampler if:	Use a Grab Sampler if:
▪ Characterization of contamination in deeper sediments is important ▪ Comparison of recent surficial vs. historical deeper sediments is needed ▪ Reduced sediment disturbance needed ▪ Reduced oxygen exposure needed ▪ Sediments are soft and fine-grained.	▪ Large sediment volumes are needed ▪ Larger grain-size or hard sediments would hamper use of a core ▪ Larger surface area of surficial sediment needed (typical for benthic invertebrate sampling).

Sampling devices such as a Ponar or VanVeen with removable or hinged bucket covers are preferred to allow access to the surface of the sediment sample without loss of water or fine-grained sediments. Key sample collection issues for grab samplers include:

- **For Sediment Grab Samples to be Subsampled in the Laboratory.** The sample should be transferred carefully and directly into a labeled container that is made of a chemically inert material, tightly sealed, and air excluded.
- **For Sediment Grab Samples to be Subsampled in the Field.** Typically, the sediment is decanted into a cleaned bowl or bag for homogenization and sub-sampling. To minimize sediment handling and artifacts of sampling for volatiles, samples may be taken directly from the grab.

5.5.2. Sediment Core Sampling Methods

Core samplers (corers) are used to characterize contamination over a depth interval that includes sub-surface sediments. They also are used to obtain sediment samples for geological characterizations and dating, investigate the historical input of contaminants to aquatic systems, and to characterize the depth of contamination at a site. Corers are an essential tool for developing three-dimensional maps of sediment contamination. Collection of subsurface layers in addition to the surface layer of sediment permits evaluation of historical or long-term impact on site conditions, and development of site-specific sediment transport mechanisms to refine the CSM.

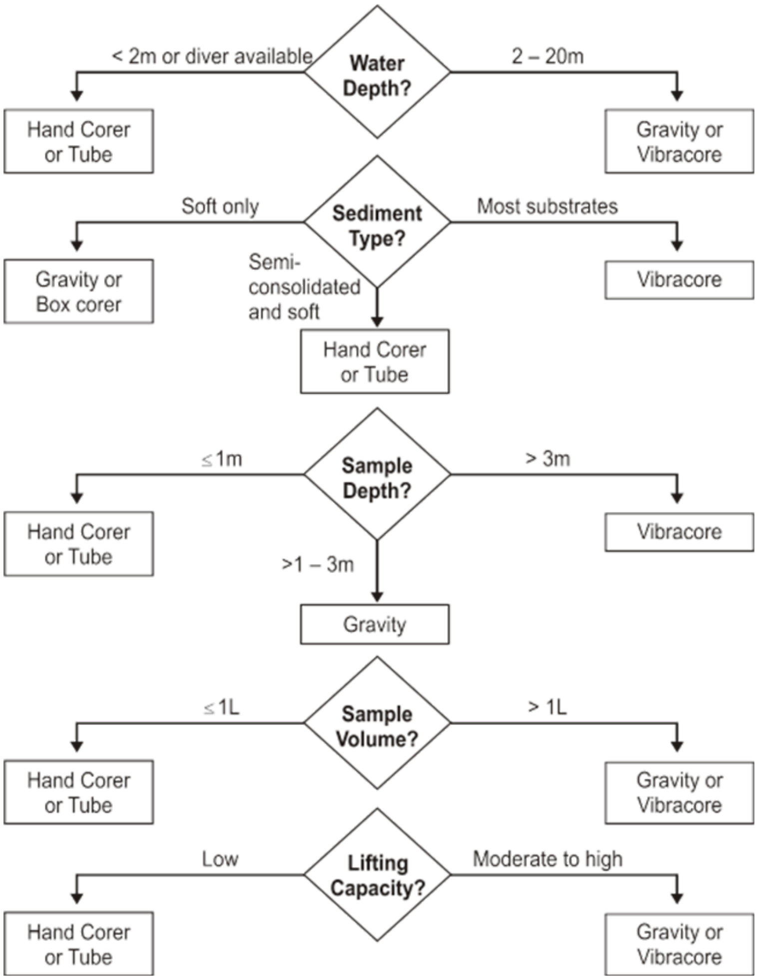
Selection of the most appropriate core sampler for a sampling event is determined based on water depth, particle size of the sediment, sediment sample depth, volume of sediment sample required for analysis, and available lifting capacity of the sampling platform/boat and winch equipment. Core samplers seldom are suitable for sampling benthic macro invertebrate samples due to the small area sampled. See Figure 5-1 for factors to consider when selecting a core sampler.

Because of the potential for a “bow wave” of sediment at the leading end of the core, resulting in compaction and inefficient flow of the sediment into the core sampler, percent recovery should be evaluated. Percent recovery is determined by dividing the internal recovery by the penetration depth. Internal recovery is the depth inside of the core tube from the top of the tube to the surface of the sediment. Core penetration is measured from the outside of the core. In recent projects in the Pacific Northwest (e.g., WindWard 2006) 75 percent core recovery has been regarded as an appropriate control limit on core recovery; however, it is recommended that project specific goals be determined.

For more information on methods for bulk sediment sampling, consult the following references:

- Standard Guide for Storage, Characterization, and Manipulation of Sediments for Toxicological Testing (Ref. 13)
- Guide for General Planning of Waste Sampling (Ref. 14)
- Guide for Core-sampling Submerged, Unconsolidated Sediments (Ref. 15)
- Standard Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates (Ref. 16)
- Handbook of Techniques for Aquatic Sediments Sampling (Ref. 17)
- Manual of Aquatic Sediment Sampling (Ref. 18)
- Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual (Ref. 1)
- Sampling for Contaminants In Sediments and Sediment Porewater (Ref. 6)
- A Compendium of Chemical, Physical and Biological Methods for Assessing and Monitoring the Remediation of Contaminated Sediment Sites (Ref. 7)
- Ocean Testing Manual Evaluation of Dredged Material Proposed for Ocean Disposal (Ref. 8)
- Inland Testing Manual: Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. (Ref. 9).

Figure 5-1. Factors in Core Sampler Selection



5.6. Bulk Sediment Handling and Analysis

If a single core will be used to represent the site, then homogenization of selected intervals and sub-sampling should occur; but if several cores will represent the site (e.g., when average conditions are desired, or greater volumes of sediment are required for biological assays), then multiple cores should be composited by depth interval. If labile chemicals, metals with a critical oxidation state (particularly organotins and mercury), acid volatile sulfides, or volatile organic compounds are of concern, a glove box to maintain anaerobic conditions during the sub-sampling activities may be needed. In addition, shielding against ultraviolet light may be needed to assure the representativeness of sediment Polycyclic aromatic hydrocarbons (PAH) samples, as some of this suite of compounds may break down. As with grab samples, inert sampling gear and mixing apparatus should be used to avoid cross-contamination. U.S. EPA's *Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses* lists additional recommendations for suitable sample containers, mixing gear, and sample preparation.

Once received at the laboratory, handling/manipulation procedures should be designed to maintain sample representativeness in terms of toxicity and chemistry by minimizing procedural artifacts. Some analytical procedures and toxicity test protocols necessitate specific manipulations for toxicity or elutriate testing.

Note: Specific analysis methods and protocols for performance of toxicity testing are beyond the scope of this document, but are available in the referenced literature.

A separate sample is usually taken for physical analysis if the sample amount is sufficient. For chemical analysis, sieving is not recommended, as it changes sediment composition (e.g., organic carbon). Sieving may be required to remove organisms that could interfere with intended biological testing. Hand-picking is recommended for debris. Should sieving be performed, it should be done for all samples to be tested, including control and reference sediments if the objective of the study is to compare results among stations.

Sediment storage for chemical analysis is typically done at <6 ºC; maximum holding times before extraction should be defined. National and regional guidance exists for maximum holding times prior to analysis.

Manipulation and storage of sediment for toxicological testing may have special requirements.

For more information on methods for bulk sediment handling and analysis, consult the following references:

- Standard Practice for Preparation of Sediment Samples for Chemical Analysis (Ref. 19)
- Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates (Ref. 20)
- Standard Guide for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphipods (Ref. 21)
- Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual (Ref. 1)
- Sampling for Contaminants In Sediments and Sediment Pore Water (Ref. 6).

5.7. Methods for Collection of Pore Water

Pore, or interstitial, water may contain contaminants partitioned from the surrounding sediment. In depositional sediments, contaminants in the pore water and in the solid phase are expected to be at thermodynamic equilibrium. This makes pore water useful for assessing contaminant levels and associated toxicity. Pore water is often isolated to provide either a matrix for toxicity testing and/or to provide an indication of the concentration and/or partitioning of contaminants within the sediment matrix. Pore water toxicity tests yield information not provided by solid-phase, elutriate, or sediment extract tests. Pore water toxicity testing has proved useful in sediment toxicity identification evaluations, and may be useful for weight of evidence regarding biological availability of some contaminants. It is critical to use the same protocol when comparing samples to one another.

In areas of known upwelling or downwelling groundwater flow through sediment, pore water may also reflect the groundwater to surface water flux conditions, and inform the CSM. Overall considerations for pore water investigations include mineralogy, subsurface flows, estimated pore water residence time, pH, temperature, redox potential, organic carbon, sulfides, and hardness (depending upon the water and metals).

Several approaches to determine pore water COPC concentrations are:

- Estimate of organics from bulk sediment concentrations (equilibrium partitioning theory)
- Estimate of metals bioavailability/toxicity via the molar ratio of acid volatile sulfides to simultaneously-extracted metals, or by the biotic-ligand model
- Traditional sediment collection followed by centrifugation or core squeezing and filtration and/or flocculation of residual particulates. The resultant supernatant (pore water) can be chemically analyzed or used for toxicity testing
- Sampling of pore water by use of suction devices, piezometers, or equipment such as the Trident System® and subsequent chemical analysis or toxicity testing
- In situ or ex situ placement of diffusion or equilibrium-based samplers directly into the sediment or extracted pore water, and measurement of either the sampler (i.e., Solid-

Phase Microextraction Devices (SPME), PE and Polyoxymethylene (POM) or the liquid contained therein (i.e., peepers). Note that some passive methods here indicated as in situ may also be applied in a laboratory setting.

Most sediment collection and processing methods likely affect pore water chemistry, contaminant bioavailability, and toxicity to some degree. “Direct” methods measure pore water chemistry and typically compare them to ambient water quality criteria or apply toxicity testing (e.g., *Daphnia* acute toxicity testing). However, relatively large volumes of water are required for toxicity testing. “Indirect” methods include peepers, dialysis bags, and passive media that can include binding agents in a bag with an ion-permeable hydrogel layer, or semipermeable membrane sorptive devices. In situ collection techniques may outperform ex situ methods for collecting pore water, as they are less subject to conditions introduced by sampling and extraction. However, in situ methods typically are restricted to waters where wading or diver activities may be applicable, and they produce relatively small volumes of pore water, which may not be suitable for the investigation needs. In situ suction devices include, push-point samplers, syringes, probes, and core-type samplers, and may be deployed primarily in soft sediments at depths to 30 centimeters.

Chemical artifacts may occur from exposure of interstitial water to oxygen. Ex situ bulk sediment centrifugation, pressurization, and suction are alternatives to in situ pore water collection, and although they may alter water chemistry, this may be necessary when larger sample pore water volumes are required, or when it is not logistically feasible to collect in situ samples.

Direct Pore Water Sampling. Small diameter piezometers may be placed in sediments where the water is shallow and the current is relatively weak. Clusters of such instruments may measure hydraulic head differences and provide pore water samples from different depths for contaminant concentration profiling. Collection can be done using peristaltic suction lift or a centrifugal or bladder pump. Suction may cause negative bias for dissolved gases and volatile organics. The pumping needs to be done at a very low rate to avoid mixing of the zones of interest. Piezometers offer advantages over diffusion samplers in that they can be sampled repeatedly, and they generally do not have volume limitations. Preservation of in situ anoxic conditions is generally not possible; however, in-line measurements of parameters such as DO and redox potential may be made to determine their approximate in situ values when suction lift is not used. Some examples of pore water samplers are:

- **Syringes.** May be used to take samples of pore water at different depths in a sediment profile in shallow or diver-accessible water depths. Pore water samples are obtained by pushing the needle to the desired depth and retracting the plunger. Syringes may not be effective in compacted sediments or gravels and can become plugged in very fine sediments. They generate relatively small volumes (up to about 0.25 L) of pore water.
- **Push-point samplers.** Useful primarily in shallow water although they can be deployed by a diver. They have a small diameter core barrel with lance tip and a “T” type handle. The small diameter barrel has holes drilled in the side at the bottom to allow water to enter. A solid plastic rod is placed in the barrel to prevent water and sediment from entering the sampler during pushing. When the sampling section of the barrel has been pushed to the target depth, the rod is withdrawn allowing pore water to enter. The water is sampled using a peristaltic pump or, in some cases, a syringe.
- **Drive-point piezometers.** Best for pore water samples at greater depth below water surface, and at greater intervals below sediment surface than push-point samplers. Drive-point piezometers may also be useful when sediment resists penetration of push-point devices. An example device configuration follows. A drive-point piezometer may be fitted to a drill rod, which is driven by an impact corer using a pneumatic hammer. The piezometer is stainless steel fitted with a bulkhead and tubing

barb, and with a screened section (typically #50 or 300 µm mesh). Pore water may be collected using a peristaltic pump connected by polypropylene tubing threaded through the hollow drill rod and attached to the barb fitting on the piezometer. The piezometer is driven to the first desired depth, and the first pore water sample collected after purging several casing volumes of water (e.g., four). If desired, following first collection, the piezometer may be driven to greater depth and another pore water sample collected following purging. For sampling at a separate location, a new, pre-cleaned piezometer should be used.

- **BAT™ sampler probe**. Consists of a tip and housing, which is sealed with a disc containing a flexible septum. The tip may be constructed of porous high-density polyethylene (HDPE) or of stainless steel to allow pore water to enter the sampler, which is under negative pressure. The stainless steel tip is driven to the desired sampling depth and a stainless steel screen is exposed that allows pore water to enter the sample housing. A tool containing an evacuated sample vial (35 – 500 mL) with a septum cap and a double-ended hypodermic needle is lowered down the push rod, and the needle penetrates the housing septum at the same time it penetrates the vial septum, thus permitting pore water to enter the vial. When the vial is full, the tool is retrieved, and the vial is stored for subsequent analysis. Porous HDPE filter tips are useful for assuring low suspended solids.
- **Trident Probe.** (Chadwick *et al.*, 2003; Chadwick and Hawkins 2008) is a hand- or diver-deployable direct push system that provides depth specific temperature and conductivity data. The on-board air hammer can drive the samplers into more compacted or stiff sediments than manual methods. It provides a better estimate where the groundwater/surface water interface is by looking at the differences in temperature and conductivity of the surface water versus water at depth, and permits selection of a sampling interval.

Following collection, pore water should be transferred to sample jars with no headspace before shipping to the laboratory at <6 ºC. *Transferring of pore water into a second jar should not be performed unless absolutely necessary*. For some chemicals, field preservation may be required.

Indirect or Passive Pore Water Samplers. These consist of bag samplers and sorbent samplers. Bag samplers include the following, and may be suitable for both inorganic and organic contaminants:

- **Peepers** and **Dialysis Bags.** In situ diffusion sampling methodologies that are suitable for shallow or diver-accessible depths. They are not standardized, and consist of small chambers with dialysis/cellulose membranes or mesh walls containing either distilled water or clean water of the appropriate salinity or hardness. The devices are buried in sediments and pore water is permitted to infiltrate the sampler. Peepers are generally limited to sediment depths of 0.2-10 cm, and generate up to 0.5 L of pore water. They require hours to several weeks to equilibrate with the sediment. There may be cross-contamination issues related to the construction materials, and biofouling may also cause artifacts. Peepers have also been used to expose organisms to sediments in situ. A possible issue with the mesh peepers is infiltration of colloidal particles into the devices.

The following are samplers that are suitable for inorganic compounds:

- **Diffusion Equilibrium in Thin Films (DET) Samplers.** Consist of hydrogel-immobilized binding agents that selectively attract ionic contaminants such as calcium, magnesium, sodium, iron, manganese, chlorine, sulfate, nitrate, alkalinity, and total CO_2. They are capable of rapid determination of flux, but they do not measure equilibrium pore water concentrations.
- **Diffusion Gradients in Thin Films (DGT).** Technique uses a similar approach to DETs to accumulate solutes on a binding agent after passage through a diffusion layer. A binding agent selective to the target ions in solution (e.g., a resin such as Chelex for metals), is immobilized in a thin layer of hydrogel separated from solution by an ion permeable hydrogel layer. Transport of ions occurs by mo-

lecular diffusion into the binding agent. Very rapidly, a steady-state linear concentration gradient is established between the solution and the binding gel, and in situ fluxes estimated.

Common features of the several of hydrophobic organic sorbent samples follow:

- Organic sorbents are designed to reach equilibrium with the freely dissolved contaminant phase in adjacent water. Quantitative results can be obtained because the sorbent typically has a known equilibrium coefficient that has been derived from laboratory calibration studies using representative compounds. Additionally, higher concentrations of contaminants occur in the sorbent, resulting in higher sensitivity than direct porewater measurements.
- The period of exposure to assure steady state depends upon the sorbent-water partitioning coefficient and volume of sorbent exposed to the pore water. High sorbent-water coefficients and large volumes of sorbent take longer for hydrophobic contaminants to equilibrate. Assurance or adjustments for steady state may be assessed (in order of preference) by loss of reference compounds with known characteristics that were introduced into the sorbent prior to placement, selection of appropriate sorbent thickness (hence volume), or by modeling contaminant transport into the sorbent.
- An advantage of some samplers is that a single device placed vertically in sediment can be subdivided into sectors that may be separately analyzed to generate a concentration profile. This use of a sorbent sampler is useful in monitoring the effectiveness of a sediment cap. The gradient of concentrations that occur can be further interpreted using advective flow modeling.
- Another advantage of sorbent samplers is that they equilibrate with truly dissolved portion of contaminants in water, hence do not measure colloidally-bound contaminants, do not require measurement of dissolved organic carbon to substantiate equilibrium partitioning calculations, and generally do not require analytical cleanup.

Note: Analytical cleanup may be required for sulfur compounds that interfere with PCB analysis.

- Passive samplers can function as biological surrogates (“biomimetics”) for a measure of bioaccumulation of hydrophobic organic compounds.

The principal hydrophobic organic sorbent devices follow:

- **Semipermeable Membrane Devices (SPMD).** Consist of flat tubular low-density polyethylene membranes containing a thin film of a high-molecular weight lipid (triolein), and depend upon diffusion of organic chemicals across a semipermeable interface to the lipid. They are used for hydrophobic organic compounds, such as PCBs, PAHs, and organochlorine pesticides. SPMDs are relatively easy to install and may have low detection and quantitation limits. They measure pore water concentrations as a time-weighted average, as opposed to an equilibrium concentration. In an aquatic environment, the uptake rate into an SPMD is linear during the first 50 days, so that the equilibrium may be estimated. An embedded tracer with a known solution rate may be applied to the SPMD prior to deployment to confirm the calculations by observing losses over time.
- **Solid-Phase Microextraction Devices (SPME).** Disposable glass fibers approximately the diameter of a human hair, coated with thin layers of poly (dimethylsiloxane) (PDMS) which sorbs organic molecules such as PAH or PCB from sediment pore water or surface water. SPME advantages include: cylindrical geometry aids insertion in sediments with small diameter cylindrical tools, low limits of quantitation, and the ability to profile contamination in sediment. SPME samplers are available that have been calibrated for PAHs at very low levels. SPME fibers are prepared by sequential sonication with hexane, acetonitrile and water, and then inserted into sediment via a push-point device with wall perforations to permit access to pore water. After equilibration, which typically takes 1 - 2 weeks, the fibers are withdrawn, cut into

lengths of interest, and transferred to autosampling vials with a small volume of solvent (e.g., 100 µL), typically hexane or acetonitrile.

There are several commercially available SPME sizes consisting of varying core size and PDMS content in the coating. Those with a 1000-µm core are sturdy and resilient for field use; those with a 210 µm core are somewhat less sturdy and harder to process in the field into autosampler vials. Sorbent coats with 30-µm layers of PDMS are high capacity and have slower uptake kinetics. A good compromise between quick uptake kinetics and ease of use and processing in the field is a 15 µm PDMS layer on 1000 µm core.

Note: Steady-state corrections are required for PCB congeners with Log K_{ow}s > 6.

- **Polyethylene (PE) Sampler**. Consists of readily-available polyethylene (e.g., 25 µm sheets) material resembling a painter's drop cloth; these may be placed into sediment or in surface water. PE samplers have been calibrated for PCBs, PAHs and other organic compounds. The rectangular geometry and pliability may require a frame for placement in situ, and direct extraction to autosampling vials is difficult. PE samplers have excellent detection limits, but also have high sorbent-water partition coefficients, which suggests longer times to steady state relative to PDMS devices.
- **Polyoxymethylene (POM) Sampler**. Similar to the PE sampler, and consists of readily-available 76 µm thick rectangular strips. POM Samplers have been used to measure sediment pore water and landfill leachate. The rectangular geometry permits placement into sediment primarily in the near-surface, and makes extraction to autosampling vials difficult. POM Samplers have excellent limits of quantitation, but high sorbent-water partition coefficients, which slow uptake kinetics relative to PDMS.

Ex situ Pore Water Techniques. Centrifugation, squeezing, and vacuum extraction/filtration are commonly used. These extraction methods may be done in the field or in the laboratory, but extraction in the laboratory just prior to analysis or testing is preferable, so that the sample is maintained as close to its original state as much as possible during transport and storage.

- Centrifugation of homogenized sediment allows collection of large volumes of pore water, and may be accomplished in an anoxic atmosphere (e.g., under nitrogen gas). Samples are typically divided among several centrifuge tubes. Centrifugation speed may have a marked influence on concentrations and toxicity. For toxicity testing, sediments should be centrifuged at 10^4 x g for 30 minutes to remove colloids. However, if toxicity testing of the interstitial fluids is the objective, this may also reduce the toxicity of the water due to the lack of colloidal contaminants. Less centrifugal force may be appropriate for contaminant studies.
- Sediment squeezing and vacuum extraction/filtration may be accomplished by relatively low-cost mechanical devices with a filter. There are also possible effects to equilibrium partitioning, internal mixing, and electrolyte changes. EPA (2001b) recommends application of moderate positive or negative pressures along with electrolyte (conductivity) monitoring during extraction. In addition, that extraction should be performed at in situ ambient temperatures to reduce alterations to interstitial water composition that may occur at temperatures different from ambient conditions.

For more information on methods for collection of pore water, consult the following references:

- Standard Practice for Preparation of Sediment Samples for Chemical Analysis (Ref. 19)
- Coastal Contaminant Migration Monitoring: The Trident Probe and UltraSeep System: Hardware Description, Protocols, and Procedures. (Ref. 22)
- Monitoring of Water and Contaminant Migration at the Groundwater–Surface Water Interface (Ref. 23)
- Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses (Ref. 1)

- Incorporating Bioavailability considerations into the Evaluation and Remediation of Contaminated Sediment Sites (Ref. 11)
- Sampling for Contaminants In Sediments and Sediment Pore Water (Ref. 6).

5.8. Benthic Macroinvertebrate Investigations

When conducting a sampling event to evaluate benthic or biological activity in aquatic sediment, specially designed sampling equipment is used to retain the biological content in the sediment. Sampling is typically limited to the upper 10 inches of the sediment layer, where benthic organisms are active.

The first part of the benthic macroinvertebrate sampling section describes methods for collection. The second part describes data acquisition and analysis.

Benthic habitats are bottom environments with distinct physical, geochemical, and biological characteristics. Benthic habitats vary widely depending upon their location and depth, and may be characterized by dominant structural features and biological communities. Estuarine and nearshore benthic habitats can be highly diverse, including shallow submerged mudflats and sandflats, rocky hard-bottom habitats, seagrass beds, kelp forests, shellfish beds, rivers, creeks, marshes, streams and coral reefs. Data on substrate type, topography, benthos, water and sediment quality, and presence or absence of toxins are all necessary to create an accurate picture of a habitat and associated ecological risk. Data from the benthos field sampling or surveys provide information on species abundance, distribution, and biomass. They may be collected by point sampling (grab or core), by tow (dredge, seine, trawl or net), by capture (cages, traps) by photographic surveys, or by other methods. Field information such as vessel type, start and end dates; investigator, and institution/agency; station numbers, positions and times; and equipment and methods are reported for each survey. Environmental data reported at each sampling site may include meteorological and sea surface conditions; surface and bottom temperature, salinity, and water quality; and sediment characteristics. Biological data would include quantity of individual organisms and total weight of organisms for each collected species.

Marine organisms are adapted to live in particular habitats, and knowledge of these sediment characteristics can provide insight into the physiology and ecology of the organisms sampled. Benthic macroinvertebrates tend to have high site fidelity and integrate long-term changes in ecosystems to inform sediment investigations of impact due to contamination. Individual macroinvertebrate species have sensitive life stages that respond to stress and integrate effects of short-term environmental variations, whereas community composition depends on long-term environmental conditions. In addition to taxonomic identification, benthic macroinvertebrate investigations may require knowledge of the feeding group to which a species belongs; for example, suspension feeders and deposit feeders. Method selection to provide for interpreting benthic invertebrate data requires knowledge of regional patterns of species abundance. Benthic community measures are often combined with chemistry and toxicity data to assess sediment impacts.

Contaminants found in marine sediments may threaten water quality, the health of aquatic organisms, and human health. Many pollutants are highly toxic and can persist in the environment for years. This can cause long-term health problems in marine invertebrates and fish such as tumors, reproductive problems, and fin rot.

Environmental contaminants may also be transferred through the food web, adversely affecting fisheries or posing a risk to human health.

The physical characteristics of the study area including water clarity, depth, circulation, currents, bottom topography, sediment quality, and study-area size influence what tools may be used effectively for macroinvertebrate surveys. Bottom characteristics may dictate the specific tools to be used for benthic investigations, as do physical characteristics of sediments. When a sediment study area is flat or gradually sloping and contains primarily fine-grained sediments, fine-scale sampling is typically preferred for defining habitat types.

As with sediment sampling, the type of benthic macroinvertebrate sampling gear depends on the nature of the substrate to be sampled. The sampling area should focus on the most predominant substrate available (in many estuaries and coastal marine areas this will be soft sediments of mud through sand grain sizes). Sampling for sediment biota is typically limited to the depth of sediment where benthic organisms are active, and this is case-specific. In slow-flowing freshwater environments, the biologically-active zone is typically shallow; however, in flowing water and estuarine/marine environments, it may be deeper.

According to U.S. EPA, numbers and kinds of macroinvertebrates collected by a particular grab are influenced by the habitat and substrate type sampled, penetration depth of the sample, the completeness of closure of the jaws (suggesting potential loss of sample during retrieval), and potential washout of surface organisms due to a shock wave associated with the sampler deployment, and stability of the sampler in particular for high water velocities.

Regional guidance may aid in developing a plan for benthic macroinvertebrates. For example, the Puget Sound Estuary Program, as applied to Puget Sound, shows the following recommendations (additional annotations in italics):

Kind of Sampler: Modified Van Veen

Area to be Sampled: 0.1 m^2

Sample Replication *(when variance is important)*: four to five per station

Sieve Mesh Size: 1.0 mm *(note, however, that Atlantic coast programs often use a 0.5-mm mesh screen)*

Initial Sieving Location: Vessel

Use of Relaxant Chemicals: Not recommended; *in some cases relaxants will reduce body distortion of soft-bodied creature; however relaxants my cause some taxa to lose limbs or other features.*

Use of Biological Stains: Rose Bengal

Level of Taxonomy: Species or Higher

Sampling Season: Variable

Recommendations for selection of benthic invertebrate samplers follow:

- Birge-Ekman, Van Veen, Ponar, and Smith-McIntyre grabs are often selected for infauna in soft substrates (river backwaters, lakes, and low-current velocity marine/estuarine conditions). Table 5-3 below summarizes characteristics of grab samplers.
- Core samplers may be used, but they sample a very small area and volume of sediment, thus representativeness of the community may be insufficient; however, this depends upon the abundance of the community, and multiple cores may be composited to provide sufficient area/volume. See Table 5-4 below for comparison of coring methods.
- For harder substrates and particularly for epifaunal invertebrates, seines and trawls may be used; however, seines and trawls typically only gather larger organisms.
- In flowing water, several techniques may be used to harvest sediment-associated biota. See Table 5-5 below for a summary of Stream Net Samplers used to collect organisms from flowing water.

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Table 5-3. Advantages and Disadvantages of Commonly Used Grab Samplers

Device	Use	Sample Depth (cm)	Sample Volume (L^3)	Advantages	Disadvantages
Orange Peel	Marine waters and deep lakes	0 – 18	10 – 20	▪ Comes in a range of sizes	▪ Need large boat, powered winch and cable line ▪ Blocking of jaws may cause sample loss
Smith-McIntyre	Deep lakes, rivers, and estuaries	0 – 4 (in deep sand)	10 – 20	▪ Reasonable quantitative samples ▪ The trigger plates provide added leverage essential to its penetration of substrate	▪ Heavy, need boat and power winch ▪ Spring-loaded jaws, hazardous ▪ Inadequate for deep burrowing organisms
Birge-Ekman, small	Lakes and marine areas; soft sediments, silt, and sand	0 – 10	≤ 3.4	▪ Handles easily without winch or crane ▪ Can be adapted for shallow water use ▪ Good for soft sediments, sand, and silt ▪ Allows sub-sampling	▪ Restricted to low current due to light weight and messenger activation ▪ May exceed target penetration depth ▪ Sub-sampling may be restricted by size of top flaps
Birge-Ekman, large	Lakes and marine areas; soft sediments, silt, and sand	0 – 30	≤ 13.3	▪ Can be adapted for shallow water use ▪ Good for soft sediments, sand, and silt ▪ Allows sub-sampling	▪ Restricted to low current conditions ▪ Penetration depth can exceed desired level due to weight of sampler ▪ Heavy, requires winch
Ponar, standard	Deep lakes, rivers, and estuaries; useful on sand, silt, or clay	0 – 10	7.25	▪ Most universal grab sampler ▪ Adequate on most substrates ▪ Large sample obtained intact, permitting sub-sampling ▪ Good for coarse and firm bottom sediments	▪ May not close completely, resulting in sample loss ▪ Metal frame may contaminate sample ▪ Heavy, requires winch
Ponar, petite	Deep lakes, rivers, and estuaries; useful on sand, silt, or clay	0 – 10	1.0	▪ Adequate for most substrates that are not compacted	▪ May not penetrate sediment to desired depth, especially in consolidated sediments ▪ Susceptible to incomplete closure and loss of sample ▪ Requires more casts to obtain sufficient sample if many analyses needed

Device	Use	Sample Depth (cm)	Sample Volume (L^3)	Advantages	Disadvantages
Van Veen	Deep lakes, rivers, and estuaries; useful on sand, silt, or clay; effective in marine environments in deep water and strong currents	0 – 30	18 – 75	▪ Adequate on most substrates that are not compacted ▪ Large sample obtained intact, permitting sub-sampling ▪ Available in stainless steel	▪ May not close completely, resulting in sample loss ▪ May close prematurely in rough waters ▪ Metal frame may contaminate sample ▪ Heavy, requires winch
Modified Van Veen (e.g., "Ted Young Grab")	Lakes and marine areas	0 – 15	≤ 18.0	▪ Fluorocarbon plastic liner can help avoid metal contamination ▪ Screened bucket cover helps reduce bow wave effects	▪ Requires winch ▪ Relatively expensive
Petersen	Deep lakes, rivers, and estuaries; useful on most substrates	0 – 30	9.45	▪ Provides large sample ▪ Penetrates most substrates	▪ Shock wave from descent may disturb fine-grained sediment ▪ Lacks lid cover to permit sub-sampling ▪ May not close completely, resulting in sample loss ▪ Metal frame may contaminate sample ▪ Restricted to low current conditions ▪ May exceed target penetration depth
Shipek, standard	Used primarily in marine waters and large, inland lakes and reservoirs; not useful for compacted sandy clay or till substrates	0 – 10	3.0	▪ Sample bucket opens to permit sub-sampling ▪ Retains fine-grained sediments effectively	▪ Metal frame may contaminate sample ▪ Heavy, requires winch ▪ Can result in loss of the topmost 2–3 cm of very fine, unconsolidated sediment
Shipek, mini	Lakes, useful for most substrates that are soft	0 – 3	0.5	▪ Handles easily without winch or crane from most platforms	▪ Requires vertical penetration ▪ Samples small volume ▪ May lose fine-grained sediment ▪ May close prematurely

Table 5-4. Advantages and Disadvantages of Commonly Used Core Samplers

(For illustrations of specific coring equipment, see http://clu-in.org/programs/21m2/sediment/)

Device/ Dimensions	Use	Sample Depth (cm)	Sample Volume (L^3)	Advantages	Disadvantages
Fluorocarbon plastic or glass tube (3.5 – 7.5 cm inner diameter (I.D.); 120 cm long)	Shallow wadeable waters or deep waters if SCUBA available; soft or semi-consolidated deposits	0 – 10	0.096 – 0.44	▪ Preserves layering and permits historical study of sediment deposition ▪ Minimal risk of contamination ▪ Rapid; samples immediately ready for laboratory shipment	▪ Small sample size necessitates repetitive sampling
Hand corer with removable fluorocarbon plastic or glass liners (3.5 – 7.5 cm I.D.; 120 cm long	Same as above except more consolidated sediments can be obtained	0 – 10	0.96 – 0.44	▪ Same advantages as fluorocarbon plastic or glass tube ▪ Penetrates substrate with greater ease through use of handles	▪ Small sample size necessitates repetitive sampling ▪ Requires careful handling to prevent spillage ▪ Requires removal of liners before repetitive sampling ▪ Barrel and core cutter metal may contaminate sample
Box corer	Same as above but the depth of the unconsolidated sediment must be at least 1 m	0 – 70	≤ 30.0	▪ Collects large, undisturbed sample; optimal for obtaining intact subsamples	▪ Difficult to handle ▪ Relatively heavy, requiring larger vessel and power winch to deploy
Gravity corer, Phlegar Corer (3.5 cm I.D., 50 cm long)	Deep lakes and rivers; semi-consolidated sediments	0 – 50	≤ 0.48	▪ Reduces risk of sample contamination ▪ Maintains sediment integrity relatively well ▪ Penetrates with sharp cutting edge	▪ Requires careful handling to avoid sediment spillage ▪ Requires repetitive and time-consuming operation and removal of liners due to small sample size
Gravity corer, Kajak-Brinkhurst Corer (5 cm I.D., 70 cm long)	Deep lakes and rivers; soft fine-grained sediments	0 – 70	≤ 1.37	▪ Collects greater volume than the Phlegar Corer	▪ Same as Phlegar Corer
Benthos gravity corer (6.6, 7.1 cm I.D., 3 m long)	Soft, fine-grained sediments	0 – 3 m	≤ 10.26	▪ Retains complete sample from tube because the core valve is fitted to the core liner ▪ Fins promote vertical penetration	▪ Requires weights for deep penetration so the required lifting capacity is 750 – 1,000 kg ▪ Requires vertical penetration ▪ Compacts sediment sample

Device/ Dimensions	Use	Sample Depth (cm)	Sample Volume (L^3)	Advantages	Disadvantages
Alpine gravity corer (3.5 cm I.D.)	Soft, fine-grained semi-consolidated substrates	≤ 2 m	≤ 1.92	▪ Allows different penetration depths due to interchangeable steel barrel	▪ Lacks stabilizing fins for vertical penetration ▪ May penetrate non-vertically and incompletely ▪ Requires a lifting capacity of 2,000 kg ▪ Disturbs sediment stratas and integrity ▪ Compacts sediment sample
Piston corers	Ocean floor and large deep lakes; most substrates	3 – 20 m	5 – 40	▪ Typically recovers a relatively undisturbed sediment core in deep waters	▪ Requires lifting capacity of > 2,000 kg ▪ Piston and piston positioning at penetration may fail ▪ Disturbs surface (0 – 0.5 m)
BMH-53 piston corer	Waters ≤ 2 m deep with extension rod; soft deposits	≤ 2 m	≤ 2	▪ Piston provides for greater sample retention	▪ Cores must be extruded onsite to other containers ▪ Metal barrels introduce risk of metal contamination
Boomerang corer (6.7 cm I.D.)	Ocean floor (up to 9,000 m deep)	1 m	3.52	▪ Requires minimal shipboard equipment so small vessels can be used	▪ Only penetrates 1.2 m ▪ Requires calm water for recovery ▪ Loses 10 – 20% of sample
Vibracorer (5.0 – 7.5 cm I.D.)	Continental shelf of oceans, large lakes; sand, silty sand, gravelly sand substrates	3 – 6 m	5.89 – 13.25	▪ For deep profiles it effectively samples most substrates with minimum disturbance ▪ Can be used in over 20 m of water depth ▪ Portable models can be operated from small vessels (e.g., 10 m long)	▪ Labor intensive ▪ Assembly and disassembly might require divers ▪ Disturbs surface (0 – 0.5 m) layer ▪ Special generator may be needed ▪ Heavier models require larger boat and power winch to deploy

Table 5-5. A Summary of Stream Net Samplers Used to Collect Organisms from Flowing Water

Net Sampler	Habitats and Substrates	Effectiveness of Device	Advantages	Limitations
Surber Stream Bottom Sampler	Shallow, flowing streams, less than 32 cm in depth with good current; rubble substrate, mud, sand, and gravel	Performance depends on current and substrate	Encloses area sampled; easily transported or constructed; samples a unit area	Difficult to set in some substrate types, that is large rubble; cannot be used efficiently in still, slow-moving streams
Portable Invertebrate Box Sampler	Same as Surber	Same as Surber	Same as Surber except completely enclosed with stable platform; can be used in weed beds	Same as Surber
Hess Sampler	Same as Surber	Same as Surber	Same as Surber except completely enclosed with stable platform; can be used in weed beds	Same as Surber
Hess Stream Bottom Sampler	Same as Surber	Same as Surber	Same as Surber except completely enclosed with stable platform; can be used in weed beds	Same as Surber
Stream-bed Fauna Sampler	Same as Surber	Same as Surber	Same as Surber except completely enclosed with stable platform; can be used in weed beds	Same as Surber
Drift Nets	Flowing rivers and streams; all substrate types	Effective in collecting all taxa which drift in the water column	Low sampling error; less time, money, and effort; collects macroinvertebrates from all substrates; usually collects more taxa	Unknown where organisms come from; terrestrial species may make up a large part of a sample in summer and periods of wind and rain; does not collect non-drifting organisms

For separating biota from sediment, standardized sampling techniques (both collection and sieve mesh size) should be followed. The use of different sieve mesh sizes for screening benthic samples limits the comparability of results between marine monitoring studies. A major advantage of using a smaller mesh size is the retention of both juvenile and adult organisms as well as large-and small-bodied taxa; however, a disadvantage is the increased time and cost of sample processing.

On-board the vessel, sieving typically occurs prior to sample preservation. If it is necessary to hold samples for later sieving, preservation proceeds sieving. When sub-sampling (sub-coring within a grab or core sample) is necessary, issues related to sub-sampling include representativeness (some benthic species have patchy distributions); and possibly damage to organisms such as worms, thereby diminishing the number of specimens that can be identified. All organisms found within the sub-core should be counted and identified.

5.8.1. Benthic Habitat Mapping

Benthic habitat mapping provides a method for relating source and distributions of contaminated sediments to sensitive or valuable aquatic resources and overall benthic habitat quality. Benthic surveys require the use of physical and biological sampling techniques. Large study areas require wide area assessments with remote sensing via hydrographic and sonar surveys to determine the physical characteristics of the benthic site without disturbing the bottom. Differing topography, sediment, and vegetation types may be resolved from sound and light reflectance patterns. Ground-truthing with finer-scale sampling methods should be used to validate interpretations of the remote-sensing data.

No single mapping method provides a comprehensive picture, so the ability to integrate, analyze, and interpret several different types of data is required in order to conduct a field-scale ecological risk assessment. Regardless of which methods are used, benthic data must be analyzed and interpreted for use in benthic habitat mapping. The analysis tools include GIS, habitat spatial analysis, and statistical or visual evaluation of benthic data and contaminant concentrations. The benthic habitat may need to be monitored over time to determine the effects of habitat change due to natural or human impacts, and to assure effective implementation of remediation strategies.

5.8.2. Toxicity Testing in Microcosms or Mesocosms

Toxicity testing of benthic macroinvertebrates, discussed briefly in Section 5.6, can complement the sediment quality and associated risk. Standard toxicity practices usually involve laboratory or field exposure chambers where organisms are exposed to natural or elevated contaminant concentration levels to assess acute and chronic risks. Attempts to obtain field data for risk assessment of contaminants released into marine/estuarine systems are often complicated by a number of factors such as complex circulation and mixing patterns, stratification, dynamic, short-term changes as well as seasonal movements of biota, and the ecosystem’s physical scale.

In recent years, microcosm and mesocosm testing has gained wide acceptance for ecological risk assessment. Microcosms and mesocosms are simulated ecosystems containing water, sediment, and communities of plants and invertebrates from field sites to be established in laboratory aquaria or in designed field systems using enclosures or caissons. Temperature, rainfall, sunlight, sediment disturbance, water circulation, bioturbation, and other natural processes can affect chemical behaviors, making it difficult to determine precisely the fate and effects of contaminants within a complex ecosystem. These simulated systems permit environmental variables to be controlled in a manner that cannot be achieved in field studies. Simulated systems may be used to evaluate higher-level ecological effects of chemicals on populations and communities. They also allow monitoring of biodegradation, natural attenuation, biota population and habitat recovery, and bioremediation.

Tests conducted in simulated ecosystems are subject to constraints that restrict the effect of physical forces, limit physical scale of the test, and introduce biases from chemical partitioning and processing along the walls of the test system. These constraints should be considered in the evaluation, but the simulated ecosystem approach still has broad application as a model of dynamic marine systems.

Mesocosms are large-scale systems that allow investigators to study and collect data over a large area. These systems are usually outdoor systems such as ponds or enclosures within a larger body of water, typically a lake, river, marsh, or ocean. For sediment ecological risk assessment purposes, the microcosm approach generally allows more efficient data collection.

Microcosm testing results are considered by most regulatory authorities to be more representative of natural conditions than results of conventional toxicity tests, which justifies the use of a smaller safety factor. For example, many aquatic populations and communities tolerate chemical exposures higher than the 50 percent lethal concentration (LC_{50}), and even sensitive test species tolerate exposures greater than the no-effect concentration (NOEC), due to factors such as population recovery or succession, water dilution, sediment transport, and other environmental factors. Microcosm results may be used to justify a higher effects threshold in these cases. Extended benthic toxicity studies can be used to justify a more accurate and less conservative assessment of ecological risk. Many regulatory authorities acknowledge that effects to the benthic community are not ecologically significant should the population recovery occur within a reasonable period of time, typically a growing season. Mirocosms have been used to demonstrate recovery in the following ways:

- Addition of spiked sediments to field sediments allows chemicals to partition between water and sediment as they would do in a natural environment; a water-sediment system may reduce toxicity by sorption which reduces the bioavailability of the compound in the water-sediment system compared to a water-only system
- Variation of chemical concentrations over time, to simulate exposure patterns typically observed in the field when chemicals disappear due to degradation or water movement
- Periodic reintroduction of invertebrates or algal organisms, which simulates natural immigration and allows observation of population recovery
- Simulation of conditions during which chemical concentrations in the water decrease over time, which may demonstrate recovery of invertebrate populations
- Benthic colonization by invertebrate species and populations may be evaluated by transferring contaminated field sediments in open tray containers to uncontaminated or reference field sites, where they are periodically monitored. Benthic colonization and recruitment from the water column may be measured by invertebrate community indices (species, abundance, diversity, evenness, dominance, richness). Benthic colonization may also be investigated by transferring contaminated sediments from the field to a flow-though, unfiltered laboratory seawater system where observations and measurements can be made more frequently.

For more information on benthic macroinvertebrate investigations, consult the following references:

- Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates (Ref. 20)
- Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates (Ref. 24)
- Standard Guide for Conducting Sediment Toxicity Tests with Polychaetous Annelids (Ref. 25)
- Estuarine and Coastal Marine Waters Bioassessment and Biocriteria Technical Guidance (Ref. 26)
- Environmental Monitoring and Assessment Program Surface Waters: Field Operations and Methods for Measuring the Ecological Condition of Wadeable Streams (Ref. 27)
- Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: *Periphyton*,

Benthic Macroinvertebrates and Fish (Ref. 28)

- Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates - Second Edition (Ref. 29)
- Methods for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with the Amphipod *Leptocheirus Plumulosus* (Ref. 30)
- Comparisons of Boating and Wading Methods Used to Assess the Status of Flowing Waters (Ref. 31)
- Logistics of Ecological Sampling on Large Rivers (Ref. 32).

5.9. References

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Chapter 6. Surface Water Sampling

6.1. Purpose

This chapter provides general guidelines and procedures for sampling approaches, methods, and equipment used for surface water sampling. Following these guidelines and procedures will greatly enhance the representativeness of the samples and accurately characterize source conditions. This chapter will help project planning and sampling personnel to evaluate available information on a sampling location, select an appropriate sampling approach, select and utilize sampling equipment, and incorporate appropriate QA/QC samples.

6.2. Scope

Surface waters are water bodies that rest or flow over land, with a surface that is open to the atmosphere. Surface water samples are collected from streams, rivers, lakes, ponds, creeks, lagoons, estuaries, surface impoundments, or coastal waters. Samples can be collected at the surface level of the subject water body or at a prescribed depth interval. Samples can be analyzed to characterize surface water quality or determine pollutant concentrations.

The procedures presented in this chapter are typical and can be modified as required to accommodate site conditions and equipment or procedural limitations. Field sampling should be conducted in accordance with appropriate federal, state, or local government compliance requirements and guidelines.

6.3. Project Planning

This section provides an overview of the planning process for conducting a surface water sampling program. Included in this section is a discussion of sampling strategy, development of a site-specific SAP, and QA considerations.

Before a surface water sampling program can begin, concise objectives should be developed regarding the intent of the sampling program. The objectives provide the framework for developing a sampling strategy for the site and preparing the SAP.

6.3.1. Sampling Strategy

The sampling strategy is designed around the objectives and begins with a review of the CSM. Refer to Chapter 2 for more information on CSM development. This model will help the Project Manager determine whether samples should be collected at the surface level or at a depth interval. The model also will help to determine whether a sample should be collected at a single location and point in time, or whether multiple samples should be collected over time, locations, or depth intervals and combined into a representative sample for analysis. From this information, the Project Manager can establish appropriate sampling locations, number and type of samples required, and an appropriate type of sampling equipment for the site conditions.

6.3.2. Site-Specific SAP

The goal of a surface water sampling program is to collect a representative sample of the current surface water conditions. The development of a site-specific SAP is the first step toward collecting a representative surface water sample. Each SAP is an instruction manual for field personnel. It should be built around the objectives and sampling strategy. See Chapter 2 of this handbook for a detailed discussion of SAP development applicable to all sampling programs.

The SAP should consider a variety of factors, including site hydrogeology, the condition and accessibility of sampling locations, suspected pollutants, DQOs, analytical methods, detection limits, surface water collection methods, sample handling procedures, and safety precautions.

The SAP cannot be implemented effectively if field personnel do not understand its contents. Many times, the primary reason that surface water sampling events are problematic is that the person preparing the SAP is not the person in the field. One method that alleviates this problem is assigning a Sampling Team Leader who is responsible for all activities in the field. The Sampling Team Leader has the appropriate level of training and experience, and will work closely with those individuals preparing the SAP to understand fully the objectives of the program. The Sampling Team Leader is responsible for all individuals in the field. If required, he or she also is responsible for providing additional training to field personnel before fieldwork begins.

6.3.3. QA

The level of data quality for each surface water sampling program, and many times for each specific sampling event, depends on the intended use of the data. It is important to remember, regardless of the level of QA/QC for any surface water sampling program, that sample integrity must be maintained during sample collection. Laboratory analysis, no matter how sophisticated, is representative only if the sample supplied to the analyst has retained its integrity.

To determine the level of quality required for each surface water sampling program or event, Site Managers and those responsible for data integrity should begin with a systematic planning process that helps define the DQOs. The DQOs clarify the study objectives, define the most appropriate type of data to collect, determine the most appropriate conditions for data collection, and specify tolerable error limits on decisions that will be used as the basis for establishing the quantity and quality of data needed to support the decision. See Chapter 2 of this manual for detailed information on QA/QC, including the systematic planning process, development of DQOs, and preparation of SAPs.

The following protocol should be used to ensure integrity and accuracy of the data collected during surface water sampling:

- The laboratory analysis should be performed by an appropriately certified or accredited laboratory for the required methodology
- All samples must be accompanied by a completed COC record
- The field blank, trip blank, equipment blank, and field duplicate samples should be collected, as appropriate, to provide for data evaluation to determine the accuracy and representativeness of surface water samples
- The field decontamination process must be followed properly to ensure QC of the field sampling
- MS/MSDs should be performed by the laboratory at a rate of one set per analyte, matrix, and preparation batch of up to a maximum of 20 samples, to validate the method selected with the matrix being analyzed
- The specific frequency of QC sample collection should be specified in the SAP, which is discussed in Chapter 2 and Appendix C.

Final data should be reviewed for correctness of numerical input, equation selection, and numerical calculations. Each site QA/QC varies with the degree of contamination, regulatory requirements, and site location.

6.4. Hazards and Safety Precautions

A site-specific HASP that addresses all anticipated hazards for each task should be prepared. The following discussion addresses common hazards associated with surface water sampling activities. Refer to Chapter 2 of this handbook for specific hazard identification techniques and detailed control methods.

When sampling surface impoundments, appropriate precautions must be employed to ensure the safety of sampling personnel. The sampling team member collecting the sample should not get too close to the edge of the impoundment, where bank failure may cause the sampler to lose his balance. The person performing the sampling should be on a lifeline and wear appropriate PPE. When performing sampling from a boat in an impoundment or flowing waters, follow appropriate boating safety procedures. In any situation where a body of water being sampled is deeper than two feet, the sampling team should wear coast guard-approved life vests.

If the surface impoundment contains known or suspected hazardous substances, the need to collect samples versus the potential risk to sampling personnel must be considered. If sampling is determined to be necessary, appropriate protective measures (e.g., use of a flat-bottomed boat for increased stability, life vests and preservers, and a back-up team) must be implemented.

When working with potentially hazardous materials, follow appropriate OSHA health and safety procedures.

6.5. Field Preparation

Surface water samples are collected from different types of water bodies with variable site conditions. Regardless of the water source, the following tasks should be completed prior to any sampling event:

- Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed
- Obtain the necessary sampling and monitoring equipment, decontaminate or pre-clean the equipment, and ensure that it is in working order
- Prepare scheduling, and coordinate with staff, clients, and regulatory agencies, if appropriate
- Perform a general site survey prior to site entry, in accordance with the site-specific HASP
- Use stakes, flags, buoys, or portable GPS coordinates to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. If collecting sediment samples, this procedure may disturb the bottom, resulting in the suspension of fine sediment particulates into the water column. To avoid sediment disturbance, river or stream sampling should start from downstream location toward upstream location.

6.5.1. Representative Sampling Considerations

To collect a representative sample, the hydrology, shape, and size of an impoundment should be determined prior to sampling. This will aid in determining the presence of phases or layers in lagoons, flow patterns in streams, and appropriate sample locations and depths.

A preliminary surface water quality survey should measure pH, temperature, and DO at points along shorelines, wetlands, creeks, and ponds. Measurements should be collected at 1meter intervals from the substrate to the surface using the appropriate monitoring instruments. For shallow waters, measurements should be taken within the top 12 inches of the water column. Measurements of pH, temperature, DO, conductivity, and oxidation-reduction potential can assist in the interpretation of analytical data and the selection of sampling sites and surface water sample collection depths.

Consider the following factors when selecting a sampling device for collecting surface waters:

- Will the sample be collected from shore or from a boat?
- At what depth should the sample be collected?
- What is the overall depth and flow direction of the river or stream?
- What type of sample will be collected (e.g., water or lagoon liquids)?

For contamination of surface water from a point source, such as an outfall or sewer line, sampling may be based on visual evidence of seepage or discharge streams. Sampling points should be established at the locations where distinct changes in pH, temperature, DO, or conductivity indicate the possible presence of contamination or leachate discharge. A GPS unit may be used to determine the coordinates of the sampling location in terms of latitude and longitude. Field equipment that should be available at the time of sampling is listed in Section 6.7.

Prior to sampling at each location, record the following information in the FLB/FN:

- Sample location, ID number, date, and water temperature at the time of collection
- pH, DO, conductivity, and depth of water (if applicable) at the sample collection location, along with any equipment calibration data
- Water depth at the midpoint or center of the water body, if applicable

- Description of site and weather conditions at time of sampling, as well as the physical characteristics of the sample.

6.5.2. Point Source Investigations

A contaminant in a discharge that feeds into a water body at a discernible, confined, and discrete conveyance is defined as a point source pollutant. An example of a point source discharge is effluent from a storm water sewer into a river through a pipe, ditch, or tunnel. Point sources are regulated under the CWA and NPDES, through permit application and approval. Sampling of these sources may be performed for characterizing discharges for permit application, with the collected data serving as a basis for establishing permit requirements for monitoring and reporting. Monitoring and analytical requirements are established by permit on a site-specific basis.

Storm water and other effluent samples that feed into a surface water body should be collected at the point source. To ensure that representative point source samples are collected in storm water investigations, specific criteria for the type of storm event that is sampled have been established. Criteria recommended by the EPA for NPDES sampling include the depth of the storm must be greater than 0.1 inch of accumulation, the storm must be preceded by at least 72 hours of dry weather, and the depth of rain and duration of event should not vary by more than 50 percent from the average depth and duration. These criteria ensure that adequate flow will be discharged, allow for build-up of pollutants during a dry interval, and ensure that the storm would be representative. State requirements may differ.

6.5.3. Nonpoint Source Investigations

A pollutant that feeds into a water body from a source that cannot be traced back to a single origin, such as storm water runoff, is termed a nonpoint source pollutant. These pollutants are monitored to determine impacts on the water quality of rivers, streams, and bays for compliance with state and local management plans.

When sampling for a nonpoint source investigation, it is important to consider special properties and precautions when developing a representative sampling design, which include:

- **Stratification.** Water body stratification occurs as a result of temperature or thermal conditions. Water temperature profile controls circulation in the water body. Warmer, less dense surface water and colder, deeper water become stratified with rapid variance of temperature and increasing depth intervals. Chemically induced stratification results when levels of a water body are separated by a steep salinity gradient. Still-water bodies, such as lakes and reservoirs, have a greater tendency to stratify than rivers or streams. One layer or zone of the water body may be exposed to a discharge point, the atmosphere, or another pathway of contamination, which can yield characteristics that are different from other layers or zones in the water body.
- **Current.** The movement, or current, in a water body can disturb mixing zones and reduce the chances of obtaining a representative sample
- **Storm Events.** Storms may turn over strata in a water body and reduce the representativeness of a sample. Precipitation or runoff may increase or reduce representative concentrations of contaminants of interest.
- **Time of Year.** Temperate water bodies experience overturn during the fall and spring season. As the air temperature warms or cools with the start of the new season, conditions in the lake change from stratified to isothermal, causing the entire water body to mix and overturn, until stratification is re-established. Time of year also influences rainy and dry periods, which affect total volume and velocity.
- **Circulation.** Movement in a water body may be the result of wind action or water density gradients. Sediment distribution may be dominated by water motion.
- **Velocity.** The speed at which a surface water body flows affects the selection of sampling locations, times, equipment, and techniques. When sampling at a river bend, velocity is the

greatest where the radius is smallest (i.e., towards the inside bank).

- **Turbidity.** Surface water can contain contaminants that adhere to the particles of suspended solids or fine particulates of sediment. These suspended sediments adhere to the water surface and will add to the water sample. Turbidity will vary due to mixing and settling in the water body.
- **Salinity.** The natural salt concentration, or salinity, of a water body may vary with proximity to the ocean, seasonal gradients, and stratification. Tidal phases of the water body must also be considered when sampling in saline waters.

For water quality investigations, surface water runoff samples should be collected at upstream and downstream locations on a subject water body. In areas where tidal influence is a consideration, a timed composite sample should be taken, with care to avoid cross-contaminating the samples. The composite sampling procedure must be based on the DQOs for data interpretation and assessment. Identify specific sampling locations and procedures in the SAP.

Additional sampling locations might be considered, depending upon the size of the site, the number of streams or rivers near the site, and the location of natural drainage swales and wetlands. If contamination of a river is suspected or documented, river or sea water levels and corresponding flow should be monitored upstream from the site and downstream from any leachate seeps or runoff. This information can be used to assess dilution effects and potential seasonal variations in contaminant concentrations due to changing water levels.

Often, the U.S. Geological Survey (USGS), state agencies, and public water supplies monitor river and stream flow and water quality at various points along major rivers or streams. Resulting data are publicly available and can be used for determining water levels, flow rates, and drainage and water quality information. Precipitation data can be acquired from local weather bureaus or the National Climatic Data Center in Asheville, North Carolina.

Nonpoint sources may feed into intermittent streams. These streams often transport contamination from a site because of surface water runoff during or after a period of heavy rainfall. If contamination is suspected because of seasonal runoff, surface water samples should be collected during and/or immediately following periods of heavy rainfall.

For investigations of compliance with storm water permit, an evaluation of the optimal sampling locations should be made. The EPA storm water program provides guidance for selecting potential sampling locations and determining the concentration of contaminants during rainfall events. It is particularly important to sample the storm flow runoff from the first 30 minutes of significant flow if not otherwise specified in the storm water permit. One sample should be collected where runoff or overflow enters the stream as well as other locations upstream of the site. Intermittent streams are not usually monitored by other agencies, so the stream depth, width, and flow rate during or after periods of heavy rainfall should be measured. The USGS can be consulted for an estimation of water drainage in particular areas.

Storm water permits often require the collection of one grab sample of constant volume, not less than 100 milliliters, every 20 minutes during the storm event, over a period of at least 3 hours. These grab samples are combined into a composite sample for analysis. Refer to Section 6.6 for more information on grab samples and composite samples. As an alternative to a time-weighted sample, a flow-weighted sample may be taken in which the volume of each grab sample to be composited is proportional to the flow rate of the discharge at the time the sample was collected.

6.5.4. Leachate

In most cases, a leachate well is installed as part of a remedial action or site characterization. Leachate wells are monitored following groundwater sampling procedures as defined in the operating permit of the facility or site.

Leachate sample collection locations should include the outfalls to shorelines. The location of leachate discharge ultimately depends on the site's physical and geological characteristics. Leachate can move laterally below ground toward a creek or stream to affect surface water quality. Samples should be collected both upstream and downstream of the site to determine the extent of contamination. In some cases, the leachate can outcrop at the top and the side of the site and flow with the surface water body. Samples should be collected not only at the leachate seeps but also upstream of them.

At each seep location, the sampler should collect samples by immersing the sample container directly in the seep. If the depth of water in the seep is not sufficient to submerge the sample container, then use a dedicated pre-cleaned container to collect the sample.

For any sample collection method used, more than one round of sampling is recommended for characterization of leachate. A minimum of two sampling events, one during a dry period and another during or immediately after precipitation, should be performed to determine variability in leachate composition. The collected samples are analyzed for any combination of the following parameters: priority pollutant organics, metals, cyanide, biochemical oxygen demand (BOD), chemical oxygen demand (COD), pH, total dissolved solids (TDS), total suspended solids (TSS), oil and grease, TOC, chloride, nitrate, phosphate, ammonia, and sulfide. All samples should be analyzed according to the permit requirements.

6.5.5. Wetland Sampling

Contaminant sources may exist around natural wetlands or other sensitive environments. Contamination from these sources can migrate from the site through surface water into wetlands, which can adsorb heavy metals and complex organics, affecting the health of the wetlands. Wetlands are defined in accordance with the *Federal Manual for Identifying and Delineating Jurisdictional Wetlands* (U.S. Fish and Wildlife Service et al., 1989).

Wetlands sampling for chemical and biota studies may be performed as part of permit applications. Composite or grab samples of surface water or sediment may be collected as described in the permit or defined in the SAP. The SAP will describe the procedures to meet the DQOs for interpreting the environmental impact of suspected contamination. When sampling in a designated wetland, it is vital to minimize the impact of the sampling event on the environment.

6.6. Sampling Procedures

Prior to initiating any field sampling activity, sampling personnel should review the HASP. All monitoring instruments and PPE should be checked thoroughly at this time. Prior to sampling at each location, water monitoring equipment probes should be decontaminated using procedures appropriate for the contaminant present and then rinsed thoroughly with ASTM Type II or better quality water.

Surface water sampling includes the collection of samples from lakes, ponds, streams, and rivers. It may be necessary to collect liquid samples from lagoons, surface impoundment, sewers, and leachate seeps. Actual sampling situations encountered in the field may vary according to the site. The most important goal of surface water sampling is to collect a representative sample of the appropriate horizons or phases present in the liquid.

Surface water can be collected either as a grab or as a composite sample. A grab sample is an individual sample collected randomly at a specific time element. A composite sample is a representative sample prepared from a combination of multiple grab sample aliquots collected at periodic time intervals or locations. A composite sample can also be a representative sample taken at a single sample point collected over an extended time interval. Grab samples collected for further preparation of a composite sample must be proportional. The time interval either between each aliquot or between the volume of each aliquot must be proportional to either the stream flow at the time of sampling, or the total stream flow since the collection of the previous aliquot. Aliquots may be collected manually or automatically using specially designed equipment. Samples that require analysis for VOAs should be submitted to the laboratory as a grab sample rather than a composited sample to minimize the potential loss of the volatile contaminants.

6.6.1. Operation of Sample Collection Devices

Sampling personnel must be properly trained in the operation of sample collection devices and should review the manufacturer's instructions prior to sample collection. Sampling operations must comply with all health and safety and QA/QC requirements, and all sampling equipment must be properly cleaned and decontaminated between each collected sample.

6.6.1.1. Laboratory-Cleaned Sample Bottle

The most widely used method for collecting surface water samples is immersion of a laboratory-cleaned sample bottle or certified pre-cleaned bottle into the water to be sampled. Alternatively, a laboratory-cleaned dipping bottle can be used to fill sample bottles if immersion of the actual sample bottle is not feasible.

Sample Bottle Use Procedures

1. Ensure the bottles are intact, with proper fitting lids with linings that are compatible with the contaminant under investigation.
2. Remove the lid, keeping it free from contamination during sampling. Immerse the bottle into the surface water to the proper depth interval and allow the water to run slowly into the bottle until it is full to zero headspace. Replace the lid.

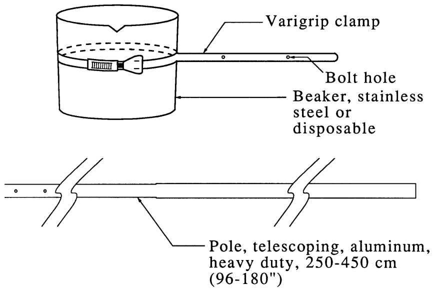
Note: Collect samples for VOC analysis first to prevent loss of volatiles due to disturbance of the water. Section 6.6.2.1 discusses collection procedures for VOC samples.

6.6.1.2. Pond Sampler

The pond sampler (also called the dipper cup sampler) is used to collect liquid samples from disposal ponds, pits, lagoons, and similar reservoirs (see Figure 6-1). The sampler consists of an adjustable clamp attached to the end of a two- or three-piece telescoping aluminum tube, which serves as the handle. Tubes can be readily purchased from most hardware or swimming pool supply stores. The clamp is used to secure a sampling beaker. The adjustable clamp and sampling beaker (stainless steel or Teflon®) can be obtained from most laboratory supply houses. The sampler can also be constructed of a polyethylene cup with a pole that is joined in a single fabrication.

Figure 6-1. Pond Sampler

QUANTITY	ITEM
1	Clamp, adjustable, 6.4 to 8.9 cm (2 1/2" to 3 1/2") for 250 to 600 ml (1/2 to 1 1/4 pt.) beakers.
1	Tube, aluminum, heavy duty, telescoping extends 2.5 to 4.5 m (8 to 15 ft.) with joint cam locking mechanism. Pole diameters 2.54 cm (1") in diameter and 3.18 cm (1 1/4") in diameter.
1	Beaker, tetrafluoropoly-ethylene or stainless steel, 250 ml (1/2 pt.).
1	Bolts 6.35 by 0.64 cm (2 1/4 by 1/4") NC.
1	Nuts, 0.64 cm (1/4") NC.



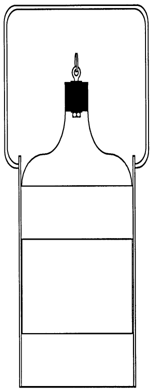
Pond Sampler Use Procedures

1. Assemble the sampler in accordance with the manufacturer's instructions. Ensure the sampling beaker or sample bottle is clamped securely to the pole.
2. Slowly submerge the beaker with minimal surface disturbance.
3. Retrieve the pond sampler from the surface water with minimal disturbance.
4. Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the dipper edge.
5. Pour the sample slowly into the appropriate sample container, allowing the sample stream to flow gently down the inside wall of the sample container with minimal entry turbulence.

6.6.1.3. Weighted Bottle Sampler

The weighted bottle sampler can be used to sample liquids from storage tanks, wells, sumps, or other reservoirs that cannot be adequately sampled with another device (see Figure 6-2). The sampler consists of a bottle (usually glass) a weight sinker, a bottle stopper, and a line used to open the bottle and to lower and raise the sampler. This sampler uses a stainless steel or carbon steel bottle basket that also serves as the weight sinker.

Figure 6-2. Weighted Bottle Sampler



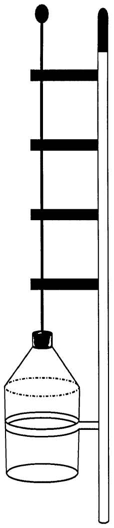
Weighted Bottle Sampler Use Procedures

1. Assemble the sampler in accordance with the manufacturer's instructions.
2. Lower the sampling device to the predetermined depth.
3. When the sampler is at the required depth, pull out the bottle stopper with a jerk of the sampler line and allow the bottle to fill completely. (This is usually when air bubbles cease.)
4. Retrieve the sampler and transfer the sample to the appropriate sample container.

6.6.1.4. Wheaton Dip Sampler

The Wheaton dip sampler is useful for collecting liquid samples from shallow areas (see Figure 6-3). The sampler consists of a glass bottle mounted on a metal pole of fixed length. Attached to the bottle's screw cap is a suction cup mounted on another metal pole.

Figure 6-3. Wheaton Dip Sampler



Wheaton Dip Sampler Use Procedures

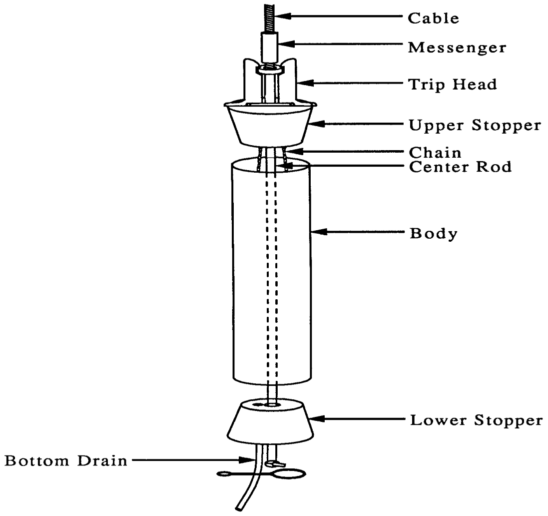
1. Assemble the sampler in accordance with the manufacturer's instructions.
2. Extend the device to the sample location, and lower to the predetermined depth.
3. Turn the metal pole attached to the suction cup to open the sample bottle.
4. When the bottle is full (usually when air bubbles cease), screw the cap back on to seal the sampling container and retrieve the sample.
5. Transfer the sample to the appropriate sample container.

6.6.1.5. Kemmerer Depth Sampler

The Kemmerer depth sampler is used to collect liquid samples from lakes, storage tanks, tank trailers, vacuum trucks, or anywhere collection depth prevents the use of other sampling devices (see Figure 6-4).

The Kemmerer depth sampler consists of an open tube with two sealing end pieces or stoppers. The end pieces can be withdrawn from the tube and set in the open position until the sampler is at the required sampling depth. Then, a weighted messenger is sent down the line or cable, releasing the end pieces and trapping the sample within the tube.

Figure 6-4. Kemmerer Depth Sampler



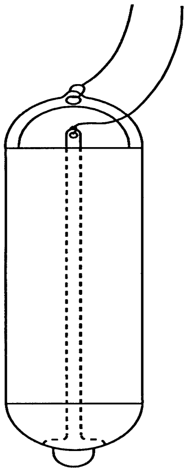
Kemmerer Depth Sampler Use Procedures

1. Set the sampler so that the sealing end pieces are pulled away from the sampling tube, allowing the water to pass through the tube.
2. Lower the sampling device to the predetermined depth. Avoid bottom disturbance.
3. Once the sampler reaches the required depth, send down the messenger, closing the sampling device.
4. Retrieve the sampler, and discharge the first 10 – 20 milliliters from the bottom drain to clear any potential contamination of the valve.
5. Pour the sample into the appropriate sample container.

6.6.1.6. Bacon Bomb Sampler

The Bacon bomb sampler is a widely used, commercially available sampler designed for sampling liquids at a depth interval (see Figure 6-5). The Bacon bomb sampler is constructed of brass or stainless steel and is available in two sizes (1.5 inches or 3.5 inches in diameter with volumes ranging from 4 or 32 fluid ounces, respectively). The sampler is equipped with a spring loaded trigger that allows liquid to enter the collection chamber when opened. When the trigger is released, liquid is prevented from flowing into or out of the collection chamber.

Figure 6-5. Bacon Bomb Sampler



Bacon Bomb Sampler Use Procedures

1. Lower the sampler carefully to the desired depth, allowing the line for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taut. This will allow the sampler to fill.
2. Release the trigger line, and retrieve the sampler.
3. Transfer the sample to the appropriate sample container by pulling upon the trigger.

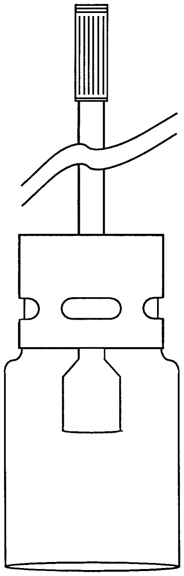
6.6.1.7. PACS Grab Sampler

The PACS grab sampler can be used to collect liquid samples from lagoons, ponds, or containers with restricted access (see Figure 6-6). For water and liquid waste sampling, the narrow neck model is useful for maintaining the collected sample inside of the device. The sampler consists of a 1,000-milliliter bottle screwed onto the end of a 6-foot fixed or telescoping handle.

PACS Grab Sampler Use Procedures

1. Assemble the sampler in accordance with the manufacturer's instructions.
2. Submerge the sampler into the liquid to be sampled.
3. Once the sampler has reached the required depth, open the sampler using the control valve at the top of the handle.

Figure 6-6. PACS Grab Sampler



4. After the sample is collected (usually when air bubbles cease), close the sample bottle.
5. Retrieve the sampler.
6. Pour the sample into the appropriate sample container.

6.6.1.8. Point Source Bailer

Point source sampling is performed to collect samples from distinct levels or points of inflow. To accomplish this task, a point source bailer can be used that yields negligible disturbance and mixing with water at different levels. This specially designed bailer has dual ball valves on both the top and bottom of the sampling chamber to isolate the sample at a discrete depth from mixing with other depth intervals.

Point Source Bailer Sampler Use Procedures

1. Assemble the sampler in accordance with the manufacturer's instructions.
2. Lower the bailer slowly to the desired sample depth on a support line. As the bailer is lowered, both ball valves are open, allowing the water to flow through the sampler.
3. On reaching the sampling depth, the bailer is raised using the support cable. The weight of the water and upward movement of the bailer keep both ball valves closed. The top ball valve prevents the sample in the bailer from mixing with water at higher levels.
4. Once at the surface, the bailer is emptied by opening the top vent and allowing the water to drain slowly through the sample release device into the sample container.

6.6.1.9. Automatic Samplers

An automated sampling system, consisting of a pump, flow sensor, distribution system, and sample containers can be used under appropriate conditions for collecting point source samples. An automated system is typically employed for collecting a composite point source sample over an extended time interval according to preprogrammed criteria. Automatic samplers can minimize labor requirements, reduce risk of human error during sampling, and provide the opportunity for remote triggering of sampling according to site conditions. Automatic sampling can be limited by cost impact from equipment and installation, and potential for crosscontamination during sampler operation. The system is not appropriate for sample collection of sources that require analysis for VOCs and chlorine, as well as microbiological agents, such as fecal coliform and *E. coli*. Automated samplers are often customized for a specific project and site conditions. Operating instructions should be developed and implemented on a project-specific basis.

Use automatic samplers when several sites are to be sampled at frequent intervals or when a continuous sample is required. Composite samplers can be used to collect time composite or flow proportional samples. In the flow proportional mode, some samplers are activated by a compatible flow meter. Refer to your specific flow meter operating manual for details on meter operation. For older models, flow proportional samples can be collected using a discrete sampler and a flow recorder, and manually compositing the individual aliquots in flow proportional amounts.

6.6.1.9.1. Installing and Programming the Automatic Composite Sampler

Use all new or pre-cleaned pump tubing each time the sampler is brought to the field and set up. Select tubing construction for the pump head and sampling train according to the analytes of interest and the allowable construction materials.

1. Cut the proper length of pre-cleaned Teflon® or Tygon® tubing.
2. Collect equipment blanks at a frequency of 5% of the samples by passing analyte-free water through the equipment that is exposed to the sample.
3. Put the collection sieve and tubing in the appropriate sample location in the wastewater stream, using conduit if necessary to hold it in place. Ensure the supporting conduit does not contaminate the incoming sample water.
4. Program the sampler per manufacturer's directions and as required in the permit or work plan conditions.
5. For a time composite sample, program the sampler to collect a minimum of 100 mL for each sample interval. Adjust the volume collected according to the duration of the sampling event, the sampling interval, and the size of the container.
6. For a flow proportional sample, program the sampler to collect a minimum of 100 mL for each sample interval, with the interval predetermined based on the flow of the waste stream.
7. Automatic Sampler Security: A lock or seal may be placed on the sampler to prevent or detect tampering. However, this procedure does not prevent tampering with the sampler tubing.

6.6.1.9.2. Sample Acquisition

1. At the end of each sampling period, stir the contents of the compositing jug (sample) and siphon contents (poured if no visible solids) into the appropriate sample containers. If the sampler was set up to collect discrete samples, ensure that the contents of each container are adequately mixed while pouring the sample into the sample container.
2. Immediately preserve the sample; if required, cap and label the sample container.

6.6.1.9.3. Long-Term Deployment of Automatic Composite Samplers

In certain sampling situations, automatic composite samplers are permanently installed at the sample stations and remain in the field for months or even years. Under these conditions, there are specific sampling issues that need to be addressed.

6.6.1.9.4. Sample Preservation

1. If the only analyte of interest is Total Phosphorus, and the project is unrelated to an NPDES permit, the sample must be chemically preserved with sulfuric acid (H_2SO_4) but does not need to be cooled to ≤6°Cwith wet ice. Other analytes will need chemical preservation and cooling.
2. The acid must be in the container prior to drawing the first composite sample into the container. When using large (i.e., 3-gallon) composite sample containers, and there is potential for the sample size to vary greatly due to variable flow rates at the site, the volume of acid for preservation should be small (e.g., 1 – 2 mLs of 50% H_2SO_4). Do not over acidify the sample. Upon sample pick-up, if needed, add additional acid to achieve the proper pH adjustment (i.e., pH ≤ 2) for preservation.
3. Cool samples with ice or refrigeration.

6.6.1.9.5. Cleaning Requirements

1. Clean composite sampler containers after collection of each composite sample using cleaning solutions and procedures.
2. Composite sample containers may be cleaned either in the field or in a fixed-base operation. Demonstrate cleaning effectiveness by collecting equipment blanks on the composite sample containers according to the frequency specified by the manufacturer. Typically, collect sampler container equipment blanks by adding a minimum of 1 L of analyte-free water to the cleaned sample container, mix the water thoroughly within the container, and then pour off an aliquot for analysis.
3. Replace tubing at a minimum of every six months. Inspect the tubing each time the composite sample container is picked up. If there is evidence of loss of elasticity or discoloration or other conditions (such as algal growth) that would impact the quality of the sample, then replace the tubing prior to the end of the six-month interval. Collect an equip-

ment blank each time new tubing is installed in the auto sampler. Collect this equipment blank by passing analyte free water through the entire length of the new tubing being deployed at each station. If the tubing is being replaced for multiple autosamplers at the same time, one equipment blank may be collected on the entire length of replacement tubing. Collect this equipment blank by passing analyte-free water through the entire length of new tubing.

6.6.1.10. Summary of Surface Water Sampling Devices

A summary of collection devices that are designed for collecting surface samples, including their advantages and disadvantages for use, are presented in Table 6-1.

6.6.2. Sample Collection Procedures

For detailed sampling procedures and preservation of samples, refer to Appendix B of this handbook. It is important to remember that COC procedures should be followed for all surface water sampling events and that all samples are to be preserved with ice to 4 ± 2ºC during both sample collection and shipment to the laboratory. Details regarding the use of COC procedures are provided in Chapter 3.

6.6.2.1. Sampling for VOCs

Use the following procedures for collecting samples for VOCs:

1. Remove the cap from a 40-millileter septum (Teflon®-lined silicon rubber) vial. Avoid contact with the inner surface.
2. Add the established amount of HCl necessary for sample preservation (if pre-preserved vials have not been supplied by the laboratory, pre-preserved vials cannot be used if the actual VOC collection container is dipped into the water being sampled).
3. Fill the vial with sample water, taking care to minimize aeration, and screw the cap on tightly to achieve zero headspace.
4. If effervescence occurs due to high carbonate salts in the samples, do not add acid preservative, and contact the Project Manager for appropriate actions such as reduced sample holding times.
5. Inspect the vial for air bubbles. If air bubbles are present, discard the sample and restart with step 1.
6. Label the vial, and place it in a resealable bag. Place the sample vials in a cooler with bagged ice sufficient to cool to 4 ± 2ºC.
7. Record all appropriate data in the FLB/FN.

6.6.2.2. Samples for SVOCs

Use the following procedures for collecting samples for SVOCs:

1. Remove the Teflon®-lined cap from a 1-liter amber glass bottle. Avoid contact with the inner surface of the cap.
2. Fill to the neck of the bottle with surface water. Add chemical preservatives, if required by the permit or method. Record the amount, type of preservative, and other preservation data per the SAP. Pre-preserved bottles also may be used for sampling surface water for extractable organics analysis, unless the sample is collected directly into the container.
3. Replace the cap tightly, attach the sample label, and place the sample bottle in a cooler with bagged ice sufficient to cool to≤6ºC.
4. Fill additional bottles by repeating steps 1 – 3.
5. Record all appropriate data in the FLB/FN.

6.6.2.3. Sampling for Additional Parameters

1. Remove the cap from the sample bottle. Avoid contact with the inner surface of the cap.
2. Fill about 90 percent of the bottle with surface water, and preserve to a pH of < 2 with nitric acid. Pre-preserved sample bottles also may be used for metals analysis.
3. Record the amount, type of preservative, and other preservation data per the SAP. Replace the cap tightly, attach label, and place the sample bottle in a cooler with bagged ice sufficient to cool to≤6ºC, if required by the permit or method.
4. Fill additional bottles by repeating steps 1 – 3.
5. Record all appropriate data in the FLB/FN.

6.6.2.4. Sampling for Additional Parameters

1. Remove the cap from the sample bottle. Avoid contact with the inner surface of the cap.
2. Fill the bottles per the SAP or method requirements.
3. Add the appropriate preservative to the samples, if required.
4. Replace the cap tightly.
5. Attach label.
6. Place sample bottles in a cooler with enough bagged ice to cool them to≤6°C, if necessary.
7. Record all appropriate data in the FLB/FN.

6.7. Sampling Equipment List

Section 3.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment that may be applicable to surface water sampling.

- Preservation chemicals and reagents
- pH meter, DO meter, conductivity meter, and thermometer
- Sample collection devices
    - Pond sampler
    - Weighted bottle sampler
    - Wheaton dip sampler
    - Kemmerer depth sampler
    - Bacon bomb sampler
    - PACS grab sampler
    - Point source bailer
    - Automatic sampler system
- Stainless steel tape and measuring rod
- Field blanks
- Trip blanks when measuring VOCs
- Cleaning materials and reagents
- Decontamination materials and detergent
- Dedicated, pre-cleaned dipping devices or bottles, if necessary.

Table 6-1. Sampling Devices for Collecting Surface water

Collection Device	Advantages	Disadvantages
Laboratory -Cleaned Sample Bottle	• Easy to use. • Low contamination risk. • No decontamination required.	• Difficult to label wet bottle. • Pre-preserved bottles cannot be used. • Exterior of bottle may contact the sample.
Pond Sampler	• Easy and inexpensive to fabricate. • Samples to depths of 3.5 meters.	• Difficult to obtain a representative sample in stratified liquids. • Difficult to decontaminate when viscous liquids are encountered.
Weighted Bottle Sampler	• Can be fabricated or purchased. • Remains unopened until it reaches sample depth.	• May be incompatible with some liquids. • Laboratory-supplied bottle may not fit into the sampler. • Mixing of the sample may occur.
Wheaton Dip Sampler	• Allows discrete samples to be taken at depth.	• Sampling depth is limited by the length of the poles. • Exterior of bottle may contact the sample.
Kemmerer Depth Sampler	• Ability to sample at various and great depths. • Allows discrete samples to be taken at depth.	• Sampling tube is exposed to water while traveling down to sampling depth.
Bacon Bomb Sampler	• Allows discrete samples to be taken at depth.	• Difficult to decontaminate. • If brass construction, may not be appropriate in metals analysis or toxicity testing. • Difficult to transfer sample to container. • The sample can become aerated.
PACS Grab Sampler	• Allows discrete samples to be taken at depth.	• Depth of the sampling is limited by the length of the pole. • Difficult to decontaminate.
Point Source Bailer	• Allows discrete samples to be taken at depth.	• May be difficult to operate. • Difficult to decontaminate.
Automated Sampler	• Allows discrete samples to be taken at depth.	• Not appropriate for collecting samples requiring VOC and microbiological analysis.

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Chapter 7. Storm Water Sampling

7.1. Purpose

Military bases and other federal facilities are regulated under the NPDES storm water program. The industrial permit program, the Municipal Separate Storm Sewer Systems (MS4) permit program, and the Construction Activities Permit Program may apply to military bases and other federal facilities. See Section 7.2 for an overview of Phase I and II storm water regulations. The following classes of discharges are regulated under NPDES storm water permit coverage:

- Operators of MS4s located in urbanized areas (UAs) or where designated
- Industrial facilities in any of the 11 categories that discharge to an MS4 or to waters of the United States. All categories of industrial activities (except construction) may certify to a condition of "no exposure" if their industrial materials and operations are not exposed to storm water, eliminating the need to obtain storm water permit coverage.
- Operators of construction activities that disturb one or more acres of land and construction sites less than one acre are regulated if part of a larger plan of development.

Storm water sampling at MS4s, industrial facilities and construction sites, including those at DoD installations, may be required to verify permit compliance. The need for storm water sampling will be determined based on the type of permit the facility obtains. For example, if a DoD industrial facility is located within a state that uses the Multi-Sector General Permit (MSGP), then analytical monitoring of outfalls associated with specific industrial sectors is required quarterly during permit years two and four. Other states may issue a general storm water permit, which requires sampling only for specific types of industrial activities. Each permit provided to the facility will outline the specific sampling and analysis requirements. This is the first step to determine:

- Sampling requirements
- The outfalls to be monitored
- The types of samples that need to be collected (e.g., grab vs. composite, first flush)
- The types of pollutants to be analyzed.

This chapter provides an overview of the:

- Federal storm water regulations including permitting requirements
- Activities at DoD facilities that may impact storm water
- Compliance monitoring strategies including sampling and analysis, QA, and health and safety requirements for storm water monitoring
- Commercially available equipment used for storm water monitoring.

7.2. Storm Water Program Overview

This section provides an overview of the federal storm water program including the differences between Phases I and II of the regulation, and permitting options for both industrial facilities and construction sites. A detailed description of the storm water regulations and permitting steps for DoD facilities can be found in the U.S. Army Environmental Center's *Storm Water Guidance Manual*, dated July 27, 2005.

7.2.1. Regulatory Summary

The CWA was amended in 1972 to prohibit discharges of any pollutant to waters of the United States from point sources[1] unless that discharge was authorized by a NPDES permit. Originally, EPA efforts to track point sources and improve water quality under the NPDES program focused on reducing pollutants in industrial wastewater and municipal sewage. Studies in the late 1980s, however, indicated that while wastewater regula-

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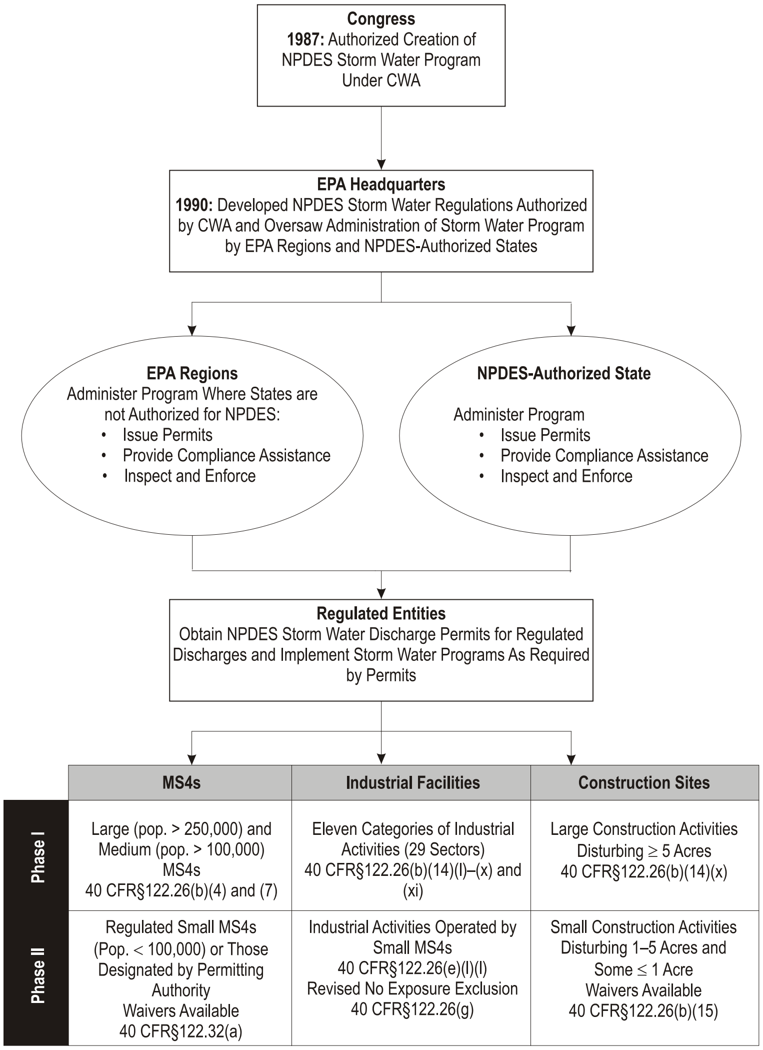
[1] A point source is defined under 40 CFR § 122.2 as "…any discernible, confined, and discrete conveyance, including but not limited to, any pipe, ditch, channel, tunnel, conduit, well, discrete fissure, container, rolling stock, concentrated animal feeding operation, landfill leachate collecting system, or vessel or other floating craft from which pollutants are or may be discharged." Since the definition of a point source is broad and can vary among individual states, installation staff should contact the appropriate NPDES permitting authority to verify a point source discharge.

tions had dramatically decreased the amount of pollutants in the nation's waters, these waters were still polluted. The major source of pollutants entering the waterways was determined to be from storm water runoff rather than from industrial or municipal discharges. Utilizing the CWA amendments of 1987, EPA promulgated NPDES Phase I storm water regulations in 1990 and Phase II regulations in 1999 to address storm water runoff from point sources (excluding agricultural runoff). These point sources include storm water runoff from MS4s, storm water discharges from certain industrial activities, and storm water runoff from construction activities. Figure 7-1 provides an overview of the NPDES storm water regulations. Specific requirements for the Phase I and Phase II rules are described below.

Additionally, Section 303(d) of the CWA, requires that states develop lists of impaired waters and establish Total Maximum Daily Loads (TMDL) for those waters. A TMDL is a calculation of the maximum amount of a pollutant that a water body can receive and still safely meet water quality standards.

Figure 7-1. Overview of NPDES Storm Water Regulations



	MS4s	Industrial Facilities	Construction Sites
Phase I	Large (pop. > 250,000) and Medium (pop. > 100,000) MS4s 40 CFR§122.26(b)(4) and (7)	Eleven Categories of Industrial Activities (29 Sectors) 40 CFR§122.26(b)(14)(I)–(x) and (xi)	Large Construction Activities Disturbing ≥ 5 Acres 40 CFR§122.26(b)(14)(x)
Phase II	Regulated Small MS4s (Pop. < 100,000) or Those Designated by Permitting Authority Waivers Available 40 CFR§122.32(a)	Industrial Activities Operated by Small MS4s 40 CFR§122.26(e)(l)(l) Revised No Exposure Exclusion 40 CFR§122.26(g)	Small Construction Activities Disturbing 1–5 Acres and Some ≤ 1 Acre Waivers Available 40 CFR§122.26(b)(15)

7.2.1.1. Phase I Regulations

The NPDES Phase I regulations require the following types of activities and systems to obtain storm water discharge permits:

- Industrial activities in 11 categories
- Construction activities disturbing five or more acres of land
- Medium MS4s (serving populations of 100,000 – 250,000)
- Large MS4s (serving populations > 250,000)
- No exposure exemption for light industry.

7.2.1.2. Phase II Regulations

The Phase II program expanded Phase I by requiring storm water discharge permits from the following activities and systems:

- Small MS4s (populations < 100,000) in UAs
- Municipal industrial facilities similar to those under Phase I
- Small construction sites disturbing one to five acres
- Construction activities disturbing less than one acre if the site is part of a larger common plan of development or sale
- Two new classes of discharges: 1) particulate matter from roof stacks and vents not otherwise regulated; and 2) visible "track out" or windblown raw materials.

This storm water sampling chapter will focus on both DoD industrial facilities and large and small construction activities at DoD installations. Information on storm water sampling at MS4s is beyond the scope of this document.

7.2.2. Permitting Requirements

A number of DoD facilities conduct industrial operations that require storm water permitting. Some of these operations include munitions manufacturing (e.g., chemical plants), metal fabrication and plating, equipment maintenance, power generation, fuel transfer and storage, and waste management areas. Regulated industrial activities at DoD facilities may be covered by an individual storm water permit or a General Storm Water Permit (GP) from either EPA or the state environmental agency. An individual permit can be more intensive, with more site-specific requirements, while the GP application process and permit requirements are usually more streamlined and less stringent. Specific permit requirements vary depending on permit type and EPA or state requirements. States with NPDES permitting authority may impose more stringent requirements or expand the scope of their programs to meet state priorities or watershed requirements.

Construction activities at DoD facilities must also comply with storm water permitting requirements. Two types of storm water permits are available for construction activities. A GP, which is typically used for construction activities, and less commonly, an individual permit. EPA and states encourage developers, construction contractors and federal facilities to apply for construction GPs when necessary, although there may be certain situations where the permitting authority would require an individual permit. For example, if the operator of a construction activity is proposing to discharge an unallowable non-storm water discharge into an impaired water body, then the permitting authority may require coverage under an individual permit.

EPA defined a regulated construction activity in the Phase I storm water rules to include:

> "…all clearing, grading, and excavation activities, except operations that result in the disturbance of less than 5 acres of total land area. Construction activity also includes the disturbance of less than 5 acres of total land area that is part of a larger common plan of development or sale if the larger common plan will ultimately disturb 5 acres or more..."

This definition was expanded by the Phase II requirements to include small construction activities that disturb one to five acres of land, or less than one acre if the construction activities are part of a common development or sale (40 CFR § 122.26(b)(15)).

Various ground-disturbing projects at DoD facilities are considered to be regulated construction activities and would require a storm water construction permit and sampling if they disturb one or more acres of land, and result in a point source discharge into waters of the United States. Typical installation construction activities may include but are not limited to:

- Clearing of an area to construct a new munitions plan
- Grading activities associated with demolition or remediation
- New road construction or full-depth reconstruction of existing roads
- Construction of housing units, offices, training facilities, or commissaries
- Combat Engineer Company training activities.

Coverage under the federal Construction General Permit (CGP) authorizes the permittee to discharge the following during construction, provided that all terms of the permit are being met:

- Storm water discharge associated with construction activities from either large or small construction sites including storm water discharge that disturbs less than one acre that is part of a larger common plan of development or sale that, when combined, disturbs one acre or more
- Storm water discharge from sites disturbing less than one acre, but designated by EPA as needing coverage under the CGP
- Storm water discharge from construction site support activities directly related to the construction site with CGP coverage (e.g., concrete or asphalt batch plants, equipment staging yards, material storage areas, track-out roadways, etc.)
- Any discharge authorized by a different NPDES permit commingled with discharge authorized by the CGP.

Additionally, the federal CGP authorizes the following non-storm water discharge, if the non-storm water component of the discharge is in compliance with Subpart 3.5 (Non-Storm Water Discharge Management) of the CGP:

- Discharge from fire-fighting and fire hydrant flushings
- Water used for vehicle and external building washing where detergents are not used
- Water used to control dust in accordance with Subpart 3.4.G of the CGP
- Potable water, including uncontaminated water line flushing
- Pavement wash water where spills or leaks of toxic or hazardous materials have not occurred (unless all spilled material has been removed) and where detergents are not used
- Uncontaminated air conditioning or compressor condensate
- Uncontaminated groundwater or spring water
- Foundation or footing drains where flows are not contaminated with process materials
- Uncontaminated excavation dewatering and landscape irrigation.

All other discharges associated with the construction activity are prohibited by the CGP and will require coverage under an individual storm water permit or another type of NPDES permit.

7.3. Storm Water Compliance

The intent of the Phase I and Phase II regulations are to prevent storm water from becoming contaminated by industrial or construction activities, rather than requiring quantitative monitoring of storm water outfalls. To implement the intent of the regulation, the control authority (EPA or the state) requires a systematic approach be taken to identify the facility’s potential storm water impacts, and to mitigate these impacts through a series of best management practices (BMPs). BMPs can be either non-structural or structural. Non-structural BMPs include procedural changes such as not allowing off-loading of fuel during rain events, covering of storm-drains during material transfer, or not storing materials outside. Structural BMPs are physical changes to the facility or implementation of technologies to prevent pollutants from leaving an industrial facility or construction site. Structural BMPs may include items such as check-dams for controlling runoff and trapping sediment, storm water retention ponds for capture of sediment, and storm drain valves to prevent releases from leaving a facility.

The first step in the systematic process to prevent contaminated storm water from leaving the industrial facility or construction site requires development of a storm water pollution prevention plan (SWPPP). The SWPPP describes issues such as potential locations for storm water contamination, possible pollutants, drainage patterns, outfalls, and BMPs that could be established to prevent polluted runoff. The storm water permit, along with information gathered during preparation of the SWPPP, provide DoD's environmental staff with the information needed to develop a compliance monitoring program for the site.

The remainder of this section discusses SWPPP development for both industrial facilities and construction sites. It provides insight into the types of pollutants expected at DoD industrial facilities and construction sites, and the current technologies and techniques being used to verify compliance through either visual monitoring or collection and analysis of storm water samples.

7.3.1. Development of a SWPPP

Preparation of a pollution prevention plan is the first step toward compliance with the storm water regulations. The SWPPP identifies the sources and types of storm water pollution associated with all industrial and construction activities covered under the GP/Individual Permit. It describes the BMPs that the installation will implement to reduce or eliminate pollutant sources and describes the monitoring requirements to verify compliance. The bullets below describe information that is provided in the primary sections of an SWPPP. See EPA's *Storm Water Management for Industrial Activities: Developing Pollution Prevention Plans and Best Management Practices*, for more guidance on preparing an SWPPP for industrial discharges.

- **Pollution Prevention Team.** The first step in developing and implementing an SWPPP is to identify a qualified individual or team of individuals who will be responsible for developing the plan and assisting with implementation. This team should represent all relevant departments in the industrial areas covered by the permit. The plan must describe the responsibilities of each team member as they relate to specific components of the plan.
- **Description of the Facility and Potential Pollution Sources.** The SWPPP must identify the activities, materials, and physical features of the regulated activity at the installation that may contribute significant amounts of pollutants to storm water runoff, or may result in pollutant discharges through a separate storm sewer system or storm water drainage system during dry weather. If there is a significant amount of pollution running onto the facility from an adjacent property, addressing or diverting this run-on should be addressed in the SWPPP. If it cannot be addressed, the permitting authority should be notified.
- **Description or Map of Site and Receiving Waters/Wetlands.** The map must include the location of all outfalls covered by the NPDES permit. It must also denote the storm water drainage patterns, locations where significant materials are exposed to rainfall and runoff, locations of major spills and leaks that have occurred in the drainage areas of the permitted outfalls within 3 years prior to receiving the Notice of Intent (NOI) to discharge storm water; and other installation-specific information.
- **Summary of Potential Pollutant Sources.** Provide a narrative description of activities, materials, and physical features of the installation that are potential pollutant sources affecting storm water quality. Describe areas of the installation that are subject to reporting under Section 313 of EPCRA.

- **Significant Spills and Leaks.** Provide a list and description of cause, type, volume, location, and response actions taken for each significant spill or leak of toxic or hazardous pollutants that occurred within 3 years prior to the date of the submission of the NOI.
- **Allowable and Prohibited Non-Storm Water Discharges.** Include a certification signed by an authorized individual to affirm that discharges from the installation have been tested or evaluated for the presence of non-storm water discharges. The certification ensures that the installation is only discharging non-storm waters allowable under the GP.
- **Selection and Implementation of Storm Water Controls.** Evaluate, select, and describe the pollution prevention measures, BMPs, and other controls that will be implemented at the installation to control storm water pollutants. The appropriateness of each selected control and how each control will address one or more of the potential pollution sources identified must be explained. This includes discussing how the individual controls relate to one another as a whole, and producing an integrated approach for preventing and controlling storm water pollution.
- **Storm Water Monitoring Requirements.** Monitoring requirements must be detailed in the SWPPP. Monitoring requirements will be detailed in the storm water permit provided by the regulatory authority and will range from visual monitoring of storm water to collection and analysis of storm water samples. The storm water monitoring section must also include a schedule that details the frequency that storm water monitoring must be performed.
- **Internal Reporting of Storm Water Information.** A reporting system must be established to ensure timely reporting of storm water management-related information to appropriate installation personnel. A description of the reporting system must be included in the SWPPP.
- **Copy of Permit.** A copy of the permit language and the confirmation letter received from the NOI processing center must be included with the SWPPP.
- **Certification.** The SWPPP must be signed and certified by the ranking officer of the installation, such as the commander or his or her duly authorized representative (an individual or a position). The representative is only duly authorized if the ranking officer documents it in writing and provides this letter to the permitting authority.

The SWPPP, along with the permit language, will help the environmental staff at a DoD facility evaluate the proper monitoring requirements for their storm water. Information collected while developing the SWPPP will help the on-site environmental staff choose the storm water monitoring locations and identify the pollutants that can be reasonably expected in storm water runoff. Information in the storm water permit provided by the control authority will help the environmental staff determine the types of monitoring that must be conducted to verify compliance (e.g., visual observations, sampling) and the frequency of monitoring.

7.3.2. Compliance Monitoring at Industrial Sites

Storm water regulated by the Phase I and Phase II rules are typically generated by either industrial or construction activities at DoD facilities. Industrial activities are identified by specific Standard Industrial Classification (SIC) codes. Other operations with no designated SIC code, such as landfills or hazardous waste treatment storage and disposal facilities, require permitting if other additional industrial activities occur at the facility are permitted. Although there is no specific SIC code that applies to all DoD facilities, the Phase I regulations state that EPA intends to address DoD facilities engaged in industrial activities as “federal facilities that meet the description of the facilities listed in the regulation” (53 FR 49416, 49432; 07 December 1988).

Table 7-1 lists some of the industrial-type activities common at DoD facilities that may require storm water permitting and possibly compliance sampling.

Table 7-1. Potential Industrial Activities at DoD Installations with Storm Water Considerations

Industrial Activity at DoD Installations	Phase I Industrial Classification	Classification Under 2000 MSGP	Chemical Sampling[a]	Additional SWPPP Reqs.[b]
Motor Pools	**(viii) Transportation facilities.** Only if related to another regulated industrial activity at the facility.	Although motor pools alone are not considered Sector P with regard to monitoring requirements, they are still considered an industrial activity and must be covered by the SWPPP if any other industrial activity requires the facility to obtain a Phase I permit.	Yes	No
Airfields	**(viii) Transportation facilities.** Only if maintenance, cleaning, or deicing activities occur.	Sector S. Installation needs permit coverage if aircraft maintenance, equipment cleaning, or deicing occurs – regardless of amount of fluid used. Deicing areas include runways, taxiways, ramps, gates, and areas where planes are deiced. Sector monitoring requirements do not apply if installation uses < 100,000 gallons of glycol-based fluid per year, or < 100 lbs. of urea/yr.	Yes	Yes
Wastewater Treatment Plants	**(ix) Treatment works**. Design flow > 1.0 million gallons per day (MGD) or are required to have an approved pretreatment program.	Sector T, Treatment Works. Applies only if unit has design flow > 1.0 MGD or is required to have an approved pretreatment program.	No	Yes
Hazardous Waste TSDFs	**(iv) Hazardous waste.** Only for facilities requiring a RCRA permit.	Sector K. Only if installation has a RCRA permit. This includes hazardous waste landfills.	Yes	No
Open Burn/Open Detonation Sites	**(iv) Hazardous waste.** Only if RCRA permitted facility.	Sector K. Only if the installation has a RCRA permit.	Yes	No
Department of Logistics Terminals/ Loading Facilities	**(xi) Light industry.**	None.	No	No
Tech Shops with Electroplating Operations	**(i) Facilities subject to effluent limitation guidelines.**	Sector AA. Produces plated ware or fabricated metal products, except for electrical-related, machinery, and transportation equipment.	Yes	Yes
Tech Shops with Metal Fabrication Operations	**(xi) Light industry.**	Sector AA. See above.	Yes	Yes
Ammunition/Ordnance Production Operations	**(xi) Light industry.**	Sector AA. See above.	Yes	Yes
Missile & Missile Parts Production	**(xi) Light industry.**	Sector AB. Produces industrial and commercial machinery or transportation equipment.	No	Yes
Aircraft Parts Fabrication	**(xi) Light industry.** If not associated with an airfield, or **(viii) Transportation**. If associated with an airfield.	Sector AB. If not associated with an airfield, or Sector S if associated with an airfield.	No	Yes
Tech Shops with Electrical	**(xi) Light industry.**	Sector AC.	No	No

Industrial Activity at DoD Installations	Phase I Industrial Classification	Classification Under 2000 MSGP	Chemical Sampling[a]	Additional SWPPP Reqs.[b]
Component/ Equip. Production				
Steam Electric Power Generating Stations	**(vii) Steam electric power generating facilities.**	Sector O. Facilities that generate steam electric power using coal, oil, natural gas, nuclear power or dual-fuel co-generation facilities.	Yes	Yes
Operations that Print & Publish DoD or Installation Documents, Periodicals, Newspapers	**(xi) Light industry.**	Sector X.	No	Yes
Misc. Service & Repair Shops, Including Electrical Shops, Furniture, Refrigeration, & Privately Owned	**None.**	None.	N/A	N/A

[a] Chemical sampling is required only if the installation meets the definition of the SIC code.
[b] The MSGP specifies additional information that must be included in the SWPPP for these sectors.

7.4. Visual Monitoring of Storm Water from Industrial Facilities

The majority of storm water monitoring at DoD industrial facilities will be audited for structural and non-structural BMPs, visual monitoring of outfalls for dry-weather flows, and visual water quality monitoring of outfalls during storm events. Visual monitoring requirements will be outlined in either the GP or the individual permit and should be included in the SWPPP, along with the locations of BMPs and storm water outfalls, drainage patterns, and potential industrial-type pollutants that could be expected based on material storage and use at the facility.

Visual monitoring of storm water outfalls during dry weather situations are conducted to determine if there are illicit connections to storm sewers that may result in the non-permitted discharge of industrial wastewater, sanitary water, non-contact cooling waters, condensates, blow down, foundation drains (i.e., groundwater) or other types of water used at the facility. If dry-weather visual monitoring discovers flow at storm water outfalls, then visual characterization of the discharge should be recorded to help identify the source.

Some characteristics that are helpful in identifying the source of the dry-weather flow include:

- Color and odor
- Turbidity
- Temperature
- Oil sheen
- Foam
- Floating particulates (e.g., paint, plastics).

If the source of the non-permitted discharge is not identified by visual observations, then collection and analysis of grab or composite samples of the dry-weather flow may be required to identify the source. Once the source of the dry-weather flow has been identified, either the illicit connection to the storm sewer must be removed or the facility must obtain a NPDES permit for discharge from the source to surface water through the storm water outfall. Figure 7-2 is a sample checklist, reproduced from EPA's NPDES *Storm Water Sampling Guidance* that can be used for dry weather visual observations.

7.5. Collection of Storm Water Samples

Data that characterize storm water discharge are valuable to permitting authorities and the permitted facility for several reasons. First, storm water sampling provides a means for evaluating the environmental risk of the storm water discharge by identifying the types and amounts of pollutants present. Evaluating these data helps determine the relative potential for the storm water discharge to contribute to water quality impacts or water quality standard violations. In addition, storm water sampling data can be used to identify potential sources of pollutants. These sources can then be either eliminated or controlled more specifically by the permit.

Industrial group or individual permitees that are required to collect samples must follow specific storm event criteria to ensure the analytical results are representative. EPA's criteria for sampling a storm event are listed below:

- The storm must result in > 0.1 inch accumulation
- The storm must be preceded by 72 hours of dry weather
- Where feasible, the depth of rain and duration of the event should not vary by more than 50 percent from the average depth and duration.

These criteria were established to: 1) ensure that adequate flow would be discharged; 2) allow for build-up of pollutants during dry weather intervals; and 3) ensure that the storm would be representative, i.e., typical for the area in terms of intensity, depth and duration. Data collected from the storm event may include the following:

- Total discharge volume
- Rainfall duration
- Maximum flow rate
- Rainfall amount
- Method of flow measurement (or estimate)
- Time from the last date that 0.1 inches of rain fell
- Date of the storm event.

Storm water samples should be taken at a storm water point source. A point source is defined as any discernible, confined, and discrete conveyance, including (but not limited to) any pipe, ditch, channel, tunnel, conduit, well, discrete fissure, container, rolling stock, concentrated animal feeding operation, landfill leachate collection system, vessel, or other floating craft from which pollutants are or may be discharged (as per 40 CFR 122.2). Included in the definition of storm water "point source" is storm water from an industrial facility that enters, and is discharged through a municipal separate storm sewer. In short, most storm water discharges can be defined as point source discharges, since they ultimately flow into some form of conveyance such as a channel or swale.

Industrial facilities with a general permit or individual permit that requires compliance sampling are usually required to collect and analyze a grab sample taken within the first 30 minutes of a storm event and flow-weighted composite samples from each of the industrial storm water point source outfalls identified in the Storm Water Pollution Prevention Plan (SWPPP). If possible, all outfalls should be sampled during the same representative storm event. The descriptions of each storm event and the outfalls sampled must be recorded.

7.5.1. Sampling Locations

The ideal sampling location would be the lowest point in the drainage area where a conveyance discharges storm water to waters of the United States or to a municipal storm sewer system. This sample point should also be easily accessible on foot in a location that will not cause hazardous sampling conditions. Ideally, the sampling site should be on the applicant's property or within the municipality's easement. If not, the field personnel should obtain permission from the owner of the property where the discharge outfall is located. Typical sampling locations may include the discharge at the end of a pipe, a ditch, or a channel, however, logistical problems with sample locations may arise (e.g., nonpoint discharges, inaccessibility of discharge point, etc.). Logistical problems with sample locations and suggested solutions are described in Figure 7-3 . In many cases, it may be necessary to locate the sampling point further upstream of the discharge point, such as in a manhole or inlet. If the storm water at a selected location is not representative of a facility's total runoff, the facility may have to sample at several locations to best characterize the total runoff from the site. In situations where discharge points are difficult to sample, the permitted facility should take the best sample possible and explain the conditions in the documentation maintained with the SWPPP.

7.5.2. Staffing Considerations

Staffing needs for sampling must be determined by the permitted facility. Factors in making the determination include the number of sample locations, the size of the area to be sampled, how far apart the locations are, the type of sampling required, the technique to be used, the number of samples to be taken (depending on how many parameters must be analyzed), and safety considerations. Sampling training is important to the success of storm water discharge characterization. Sampling conducted by untrained personnel may result in data that is unrepresentative of the individual facilities storm water discharge. This data may be rejected by a permitting authority, which would then require another sampling effort.

Figure 7-2. Checklist for Conducting Dry Weather Evaluations

1. Date of Inspection: _________________
2. Facility name and address: _____________
____
____
____
____
3. End Date of last rain event: __________
4. Inspector name: ____________________
5. Type of outfall
☐ Concrete ☐ Pipe ☐ Grassed
☐ Rock ☐ Other

6. Is there viable flow from the pipe?
☐ Yes ☐ No

If yes, check all that apply. If no, go to number 7.

☐ Colored water (describe) _________
☐ Odor* (describe) ________________
☐ Murky
☐ Floating objects (describe) ________
☐ Absence of plant life surrounding conveyance
☐ Scum

☐ Oily sheen
☐ Sludge present
☐ Clear water
☐ Stains on conveyance
☐ Notable difference in plant life surrounding conveyance
☐ Suds ☐ Other: _________________

*e.g., rotten eggs, earthy, chemical, chlorine, soap, putrescence, gasoline, musty, etc.

Estimate the flow either visually or by describing the width, height, and shape of the conveyance and the approximate percentage of the conveyance where flow is present or the approximate depth of the flow. Describe your estimate.

7. Is there standing water present?
☐ Yes ☐ No

If yes, check all that apply. If no, go to number 8.

☐ Colored water (describe) _________
☐ Odor* (describe) ________________
☐ Murky
☐ Floating objects (describe) ________
☐ Absence of plant life surrounding conveyance
☐ Suds

☐ Oily sheen
☐ Sludge present
☐ Clear water
☐ Stains on conveyance
☐ Notable difference in plant life surrounding conveyance
☐ Scum ☐ Other: _________________

*e.g., rotten eggs, earthy, chemical, chlorine, soap, putrescence, gasoline, musty, etc.

8. From the inspection locations, can you see any unusual piping or ditches that drain to the storm water conveyance?
☐ Yes ☐ No
9. Is there any overload flow visible from the discharge location? ☐ Yes ☐ No
10. Are there dead animals present?
☐ Yes ☐ No

Signature:

Figure 7-3. Solutions to Sampling Location Problems

Problem:	Sampling where storm water commingles with process or non-process water.
Solution:	Attempt to sample the storm water discharge before it mixes with the non-storm water discharge. If this is not possible, sample the discharge during dry and wet weather and present both sets of data to the permitting authority. This will provide an indication of the contribution of pollutants from each source.
Problem:	Numerous small point discharges.
Solution:	Impound channel or join flow by building a weir or digging a ditch to collect discharge at a low point for sampling purposes. This artificial collection point should be lined with plastic to prevent infiltration and/or high levels of sediment. Or, sample at several locations to represent total site runoff.
Problem:	Inaccessible discharge point, such as underwater discharges or unreachable discharges (e.g., out of a cliff).
Solution:	Go up the pipe to sample, i.e., to the nearest manhole or inspection point. If these are not available, tap into the pipe or sample at several locations to best represent total site runoff.
Problem:	Managing multiple sampling sites to collect grab samples during the first 30 minutes (industrial facilities only).
Solution:	Have a sampling crew ready for mobilization when forecasts indicate that a representative storm will occur, or sample several different representative events. For most parameters, automatic samplers may also be used to collect samples within the first 30 minutes triggered by the amount of rainfall, the depth of flow, flow volume or time.
Problem:	Commingling of parking lot runoff with discharge associated with industrial activity.
Solution:	The combined runoff must be sampled at the discharge point as near as possible to the receiving water or the parking lot drain inlet if there is one.
Problem:	Sampling in manholes.
Solution:	Sample in manholes only when necessary, as this requires training on confined space entry.
Problem:	Run-on from other property.
Solution:	If possible, estimate the volume of off-site run-on contributions and off-site run-on sources of pollutants to perform a mass balance calculation. Include this information in the permit application. If this estimation is not possible, provide a narrative discussion of the upstream site (e.g., is it developed, if so the type of facility, the types of pollutants that may be present on the site, etc.).

7.5.3. Sample Type: Grab and Composite Storm Water Samples

Because of the variable nature of storm water flows during a rainfall event and different analytical considerations for certain pollutants, the storm water regulations establish specific requirements for sample collection techniques. To comply with storm water requirements, the sample type (grab or composite) must be collected in accordance with 40 CFR 122.21(g)(7) and 40 CFR Part 136. Definitions of grab and composite storm water samples are listed below.

- **Grab Sample.** A grab sample is a discrete, individual sample taken within a short period of time, usually < 15 minutes. Analysis of grab samples characterizes the quality of a storm water discharge at the given time of the discharge.
- **Composite Sample.** A composite sample is a mixed or combined sample that is formed by combining a series of individual and discrete samples of specific volumes at specified intervals. Although these intervals can be time-weighted or flow-weighted, the storm water regulations require the collection of flow-weighted composite samples. This means that discrete aliquots or samples are collected and combined in proportion to flow volume rather than time. Composite samples characterize the quality of a storm water discharge over a longer time period, such as the duration of a storm event. Flow-weighted composite samples must be collected during the first 3 hours of discharge or the entire discharge (if it is < 3 hours) for industrial facilities.

Storm water permits clearly specify which pollutants must be analyzed by grab sample and which by composite sample. Although the requirements in 40 CFR 122.21(g)(7) do not explicitly specify either manual or automatic sampling techniques, the approved analytical methods contained in 40 CFR Part 136 direct that grab samples must be manually collected for certain pollutants. Table 7-2 provides a list of the typical pollutants that are required for storm water permit compliance, and the type of sample (grab or composite) that must be collected. The actual list of pollutants for each facility will be included in the storm water permit. The advantages and disadvantages of using manual versus automatic sampling techniques for storm water samples are provided in Table 7-3.

7.5.4. Storm Water Sampling from Retention Ponds

If storm water from a facility discharges after flowing through a retention pond or other treatment system, sample as the storm water flows out at the discharge point. Ponds may hold storm water for a time before discharge begins. Sample within the first hour, preferably 30 minutes from when the pond begins to discharge.

Retention ponds with greater than a 24-hour holding time for a representative storm event may be sampled by grab sample. Composite sampling may not be necessary. The rationale for this is because the water is held for at least 24 hours, a thorough mixing can occur within the pond. Therefore, a single grab sample of the effluent from the discharge of the pond may represent a composite of the storm water contained in the pond. However, if the pond does not thoroughly mix the discharge, thereby compositing the sample, then a regular grab and a composite sample taken at multiple times should be taken at the inflow to the pond. Since each pond will vary in its capability to composite the water, permittees must carefully evaluate whether the pond is thoroughly mixing the discharge.

Factors such as pond design and maintenance are important in making this evaluation. For example, poor pond design, where the outfall and inflow points are too closely positioned, can cause short-circuiting and inadequate mixing. In addition, poor maintenance may lead to excessive re-suspension of any deposited silt and sediment during heavy inflows.

Because of these factors, the permittee should determine the best location to sample the pond (e.g., at the outfall, at the outfall structure, or in the pond) to ensure that a representative storm water sample is taken. If adequate compositing does not occur within the pond, the permittee should conduct routine grab and flow-weighted composite sampling.

Table 7-2. Typical Storm Water Pollutants, Possible Analytical Methods, and Sample Collection Techniques

Storm Water Pollutant	Possible Analytical Method	Sample Technique
pH	Field measurement	Grab
Temperature	Field measurement	Grab
Oil and Grease	EPA 1664	Grab
TSS	EPA 160	Flow-Weighted Composite
BOD_5	EPA 405	Flow-Weighted Composite
COD	EPA 410	Flow-Weighted Composite
Nitrate and Nitrite Nitrogen	EPA 353	Flow-Weighted Composite
Ammonia Nitrogen	EPA 350	Flow-Weighted Composite
Fecal Coliform	Standard Methods 9222	Grab
Fecal Streptococcus	Standard Methods 9230	Grab
Total Phosphorous	EPA 365	Flow-Weighted Composite
Priority Metals	EPA 200, 200, 200, and 245 (Mercury Only)	Flow-Weighted Composite
Total Cyanide	EPA 335	Flow-Weighted Composite
Total Phenolics	EPA 420	Flow-Weighted Composite
VOCs	EPA 1624	Grab
SVOCs	EPA 1625	Flow-Weighted Composite
Pesticides	EPA 1657	Flow-Weighted Composite

Table 7-3. Comparison of Manual and Automatic Sampling Techniques for Collection of Storm Water Samples

Sampling Technique	Advantages	Disadvantages
Manual Grabs	Appropriate for all pollutants Minimum equipment required	Labor intensive Environment possibly dangerous to field personnel May be difficult to get personnel and equipment to the storm water outfall within the 30 minute requirement Potential for human error
Manual Flow-Weighted Composites (multiple grabs)	Appropriate for all pollutants Minimum equipment required	Labor-intensive Environment possibly dangerous to field personnel Human error may have significant impact on sample representativeness Requires flow measurements taken during sampling
Automatic Grab Samplers	Minimizes labor requirements Reduced risk of human error Reduced personnel exposure to unsafe conditions Sampling may be triggered remotely or initiated according to present conditions	Samples collected for oil and grease (O&G) may not be representative Automatic samplers cannot collect samples for VOCs analysis Costly if numerous sampling sites require the purchase of equipment Requires equipment installation and maintenance Requires operator training May not be appropriate for pH and temperature measurement May not be appropriate for parameters with short holding times (e.g., fecal streptococcus, fecal coliform chlorine)

Sampling Technique	Advantages	Disadvantages
		Cross-contamination of aliquot if tubing/bottles not properly washed
Automatic Flow-Weighted Composite Samplers	Minimizes labor requirements Reduced risk of human error Reduced personnel exposure to unsafe conditions May eliminate the need for manual compositing of aliquots Sampling may be triggered remotely or initiated according to on-site conditions	Not acceptable for VOCs sampling Costly if numerous sampling sites require the purchase of equipment Requires equipment installation and maintenance, may malfunction Requires initial operator training Requires accurate flow measurement equipment tied to sampler Cross-contamination of aliquot if tubing and bottles not properly washed

7.6. Obtaining Flow Data

In addition to collecting samples of storm water discharges, permittees must collect data characterizing the flow rate and flow volume for each storm water discharge sampled. Flow rate is the quantity of storm water discharged from an outfall per unit of time. Total flow is a measure of the total volume of storm water runoff discharged during a rain event. Flow rate and volume either can be measured specifically or can be estimated, based on rainfall measurements, velocities, and depth of flows. To collect flow-weighted composite samples, flow rate data is necessary to combine proportional volumes of individually collected aliquots. Permittees must also report the mass of pollutants contained in storm water discharges. To determine mass loadings of pollutants, applicants must measure both discharge flow rate and pollutant concentration. This section presents methods for obtaining flow data.

7.6.1. Measuring Flow Rates

Flow rates for storm water discharges are most accurately measured using primary flow measuring devices such as weirs, simple flumes, and Palmer-Bowlus flumes. Weirs consist of a crest located across the width of an open channel (located at a right angle to the direction of the flow). The flow of water is impeded, causing water to overflow the crest. Weirs are inexpensive and particularly valuable in measuring flow in natural or manmade swales because they are easily installed in irregular shaped channels. When flow exceeds the capacity of the weir and water overtops the weir crest, flow depth actually diminishes as the water approaches the weir and flows cannot be measured accurately. Figure 7-4 is a diagram of a weir system showing the method to calculate flow through a weir.

Flumes are structures that force water through a narrow channel. They consist of a converging section, a throat, and a diverging section. Figure 7-5 is a diagram of a flume and flow rates through the flume. Flumes have fixed specifications relating to geometric shape; they vary only in throat width. Due to these geometric constraints, flumes may be expensive to install. They typically are used in permanent flow measurement points and are most commonly placed in concrete-lined channels. However, flumes can also be used in temporary points. Flumes can provide accurate measurements for a relatively wide range of flow rates.

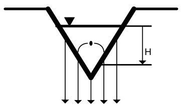
Palmer-Bowlus flumes are also used for flow measurement at some facilities. Palmer-Bowlus flumes are designed for installation in an existing circular channel (e.g., a manhole channel) and are available as portable measurement devices. Figure 7-6 is a diagram of a Palmer-Bowlus flume flow measurement system. While Palmer-Bowlus flumes are inexpensive, self cleaning, and easy to install, they can only measure accurately over a narrow range of flow rates. The flow from a Palmer-Bowlus flume is calculated using the height between the floor of the flume portion and the water level, not the total head of the water level.

Where flow measurement devices are not already installed, portable devices should be considered.

There are many permanent and portable types of flow measurement devices available. Proper analysis of site discharge conditions must be conducted prior to purchase and implementation of these devices.

Figure 7-4. Weirs

V-Notch

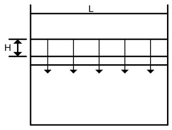


$Q = 2.5\,H^{2.5}$ (90°)
$Q = 1.443\,H^{2.5}$ (60°)
$Q = 1.035\,H^{2.5}$ (45°)
$Q = 0.676\,H^{2.5}$ (30°)
$Q = 0.497\,H^{2.5}$ (22½°)

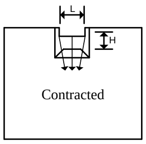
Q = Flow Rate

H = Depth of flow (Head)

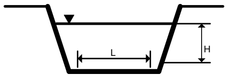
Rectangular (without contractions) $Q = 3.33\,L\,H^{1.5}$



Rectangular (with contractions) $Q = 3.33\,(L - 0.2\,H)^{1.5}$



Cipolleti (trapezoidal) $Q = 3.367\,b - H^{1.5}$

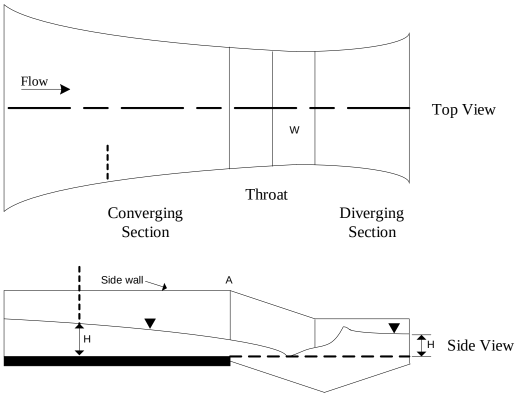


Source: Civil Engineering Reference Manual. 5th Edition, by Michael R. Lindeburg, PE, with permission from the publisher, Professional Publications, Inc., Belmont, California, 1989.

Figure 7-5. Flumes

Parshall Flume

$Q = 0.338\,H^{1.55}$ (1 inch)
$Q = 0.676\,H^{1.55}$ (2 inches)
$Q = 0.992\,H^{1.547}$ (3 inches)
$Q = 2.09\,H^{1.55}$ (6 inches)
$Q = 3.07\,H^{1.55}$ (9 inches)
$Q = 4\,W\,H^{1.522}\,W^{0.26}$ (1-8 feet)
$Q = (3.6875\,W + 2.5)H^{1.6}$ (10-50 feet)
Q = Flow rate
H = Depth of flow (Head)



Source: Civil Engineering Reference Manual 5th Edition, by Michael R. Lindeburg, PE
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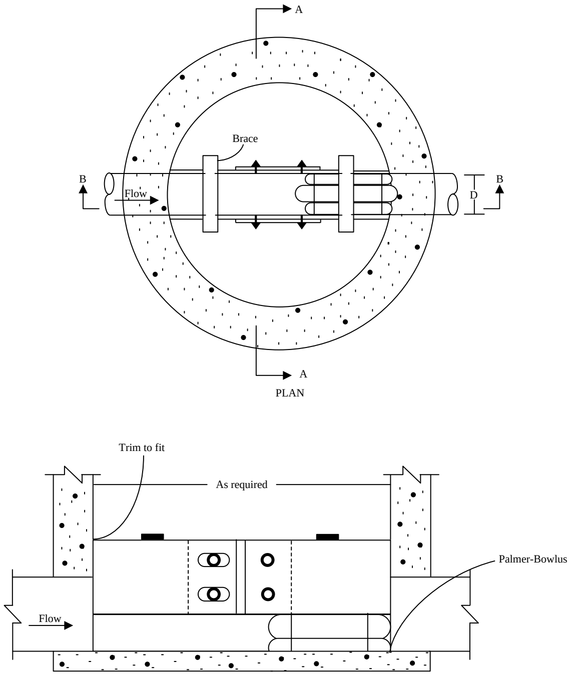
Figure 7-6. Palmer-Bowlus Flume



Source: Wastewater Engineering: Treatment, Disposal, Reuse. 2nd Edition, Metcalf & Eddy, Inc., with permission from the publisher, McGraw-Hill Book Co., New York, 1979.

7.7. Compliance Monitoring at Construction Sites

CGPs may require that a visual inspection program be implemented to periodically evaluate the integrity of the BMPs and visually observe and note the condition of storm water leaving the construction site. CGPs may also require that a SAP be developed and conducted for pollutants which:

- Are not visually detectable in storm water discharges
- Are known to occur on the construction site
- Could cause or contribute to an exceedance of water quality objectives in the receiving water.

Pollutants that should be considered for inclusion in the sampling and analysis strategy are those identified during development of the SWPPP. The CGP requires that the SWPPP identify a strategy for conducting the sampling and analysis, including the frequency and location(s) at which sampling will be conducted. Sampling for non-visibly detectable pollutants is usually required under the following two conditions:

- Visual inspections, currently required before, during, and after storm events, indicate that there has been a breach, malfunction, leakage, or spill from a structural BMP that could result in the discharge of pollutants in storm water and the pollutants would not be visually detectable
- Storm water encounters soil amendments, other exposed materials, or site contamination that is discharged at a location off of the construction site.

A sample of uncontaminated (background) storm water from the site must be collected for comparison with the sample(s) collected from storm water suspected of containing construction-related pollutants. The CGP may also state that the SWPPP needs to describe the sampling procedure, location, and rationale for obtaining the uncontaminated sample of storm water.

7.7.1. "Known or Should Be Known Pollutants" at Construction Sites

Pollutants can be considered "known" or "should be known" to occur on the construction site if they are currently in use or are present because of previous land uses. These may include the following materials:

- Used in the construction activities
- Stored on the construction site
- Spilled during construction operations and not cleaned up
- Stored or used in a manner that presented the potential for a release of the materials during past land use activities
- Spilled during previous land use activities and not cleaned up
- Applied to the soil as part of past land use activities.

Construction material inventories and the project SWPPP should provide adequate information on materials currently in use or proposed for use on the construction site. Dischargers should review existing environmental and real estate documentation to determine the potential for pollutants to exist on the construction site because of past land use activities. Potential sources of information for previously existing contamination and past land uses include environmental assessments, initial studies, environmental impact reports or environmental impact statements prepared under the requirements of NEPA, and Phase 1 assessments prepared for property transfers. In some instances, the results of soil chemical analyses may be available and can provide additional information on potential contamination.

7.7.2. Deciding When Sampling Is Required at Construction Sites

All construction projects must ensure that proper inspections are conducted throughout the duration of the project to make sure that appropriate BMPs have been implemented, are being maintained, and are effective in preventing potential pollutants from coming in contact with storm water and causing or contributing to an exceedance of water quality objectives in the receiving waters.

The frequency of sampling for non-visible pollutants must be determined based on the exposure of pollutant sources. Runoff only needs to be sampled when there is exposure of a pollutant source to storm water that enters a storm drain or surface water. Inspections of material storage areas that identify a non-structural or structural BMP failure that exposes potential non-visible pollutants to storm water that runs off the construction site will trigger a requirement for sampling and analysis. If spills are cleaned up and the contaminated material is isolated, eliminating exposure to storm water runoff, then sampling is not necessary. For instances when the potential for previously existing contamination is identified, it may be appropriate to conduct screening analysis during the first one or two storm events of the season to determine if the potential contaminant is running off the construction site. Figure 7-7 provides a flow chart to help determine when sampling and analysis is required.

7.7.3. When Sampling and Analysis May Not Be Required at Construction Sites

Sampling and analysis is typically not required under the following conditions:

- Where a construction project is "self-contained," meaning that the project generates no runoff or any potential runoff discharges containing pollutants, or pollutants can be totally contained within the construction project site without discharging to a water body or storm drain system
- Where construction materials and compounds are kept or used so that they are not in contact with storm water (e.g., in water-tight containers, under a water-tight roof, inside a building, etc.)
- Where for specific pollutants, the BMPs implemented at the construction site fully contain the exposed pollutants (e.g., bermed concrete washout area)
- For building materials that are in their final constructed form or are designed for exposure (e.g., fence materials, support structures and equipment that will remain exposed at the completion of the project, etc.)
- Where pollutants may have been spilled or released on site, but have been properly cleaned up and storm water exposure has been eliminated prior to a storm event
- For stockpiles of construction materials for which both cover and containment BMPs have been properly implemented to protect them from run-on and from contributing pollutants to storm water runoff.

However, a contingency sampling strategy should be prepared in the event of an incidental discharge. The SWPPP should also describe why sampling and analysis is not expected to be needed.

7.7.4. When Sampling and Analysis May Be Required at the Construction Site

Sampling and analysis is required when non-visible pollutants have the potential to contact storm water and run off the construction site into a storm drainage system or water body. Examples of this situation follow:

- Where construction materials and compounds are stored or used such that they may encounter storm water
- For construction projects that utilize soil amendments that can encounter storm water runoff. (If independent test data are available that demonstrate acceptable concentration levels, sampling and analysis may not be required. Contact the appropriate regulatory authority to determine acceptable concentration(s) of the material(s) in question.)
- When a leak or spill occurs prior to a storm event and is not fully contained and cleaned up
- When a leak or spill occurs during a storm event and cannot immediately be isolated or cleaned up, and the possibility of an off-site discharge exists
- When, during regular inspections of stockpiles, it is discovered that cover and containment BMPs have been compromised and storm water encounters the stockpiled materials resulting in runoff discharging into a storm drain system or water body

- When material storage BMPs have been compromised, breached, or have failed. If determined that sampling is needed, storm water runoff samples must be collected regardless of the time of year, status of the construction site, or day of the week. Samples should be collected during the first 3 hours of runoff. Storm water inspections and sample collections are required even during weekends or holidays.

A list of potential pollutants must be developed based on a review of potential sources from the SWPPP. It should include construction related materials, soil amendments, and historic contaminants. Identify from this list those pollutants that are not visibly detectable. These constituents will likely require sampling and analysis of runoff if the materials are exposed to storm water. Consult with an analytical laboratory or water quality chemist to determine if there are field tests or indicator parameters that can be used. Table 7-4 is a list of typical construction materials that might cause non-visible contamination of runoff if exposed to storm water.

7.7.5. Deciding Where to Sample at a Construction Site

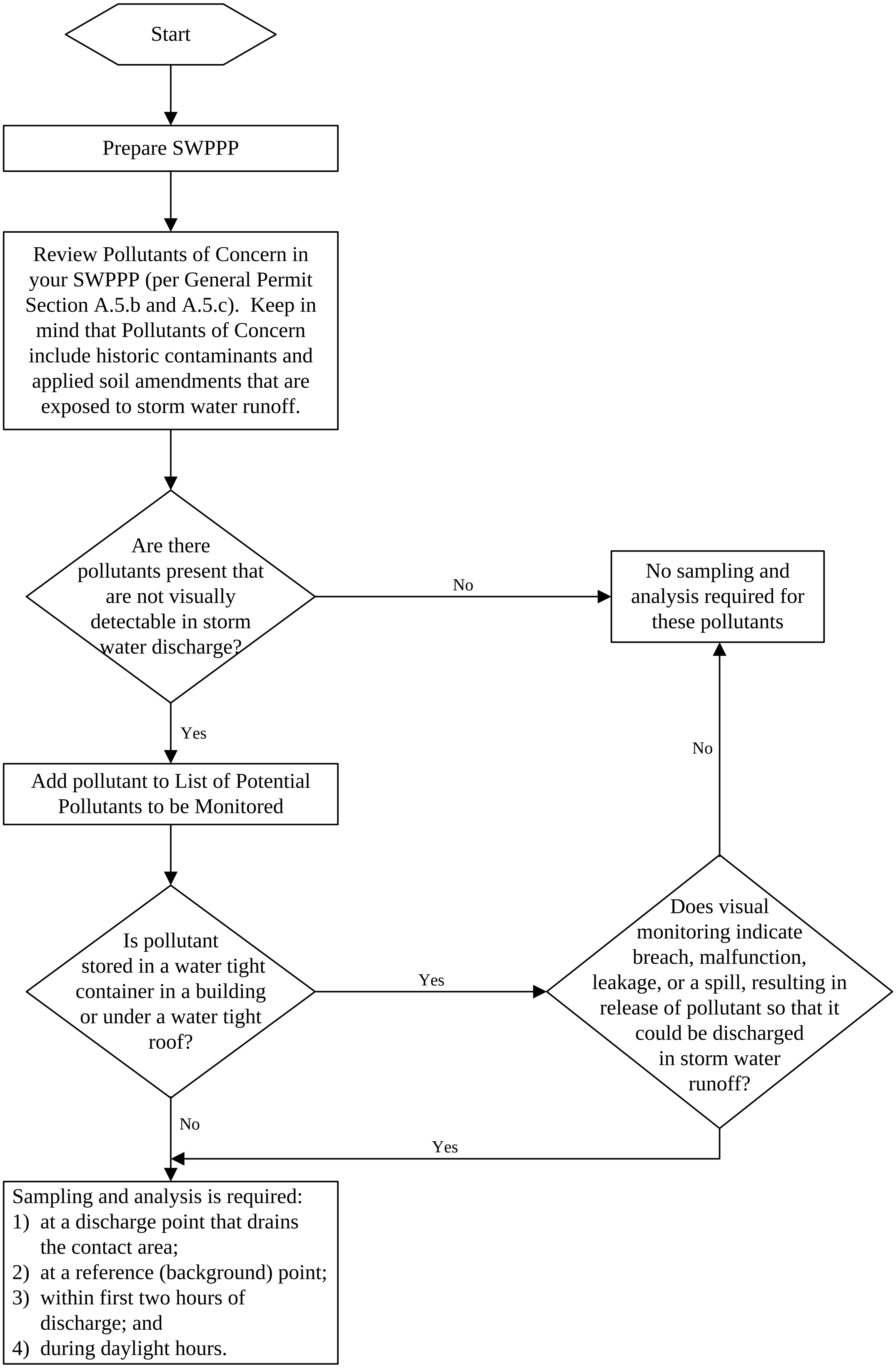
Sampling locations must be identified that provide information on both the runoff quality that is affected by material storage, historic contamination or other exposed potential pollutants, and the background runoff quality (i.e., uncontaminated sample). Material storage may be confined to a small area of the project site while historic contamination or exposed materials such as soil amendments, may be widely spread throughout the construction site. For this reason, the sampling locations identified for these two types of potential pollutants may be different.

Samples must be collected at locations identified in the SWPPP or areas identified by visual observations or inspections where there has been a structural BMP failure or breach that can be safely accessed. A location that is not affected by material storage activities or by runoff from material storage areas should be selected as a background or reference sampling location for collecting the uncontaminated runoff sample. For a widely spread potential contaminant, sampling locations may need to be selected at the perimeter of the site, where storm water enters (unaffected by site activities) and leaves (affected by site activities).

Table 7-4. List of Common Potential Non-Visible Pollutants

Category	Potential Pollutant Source	Field Indicator of Pollutant Release	Laboratory Analysis
Line Flushing	Chlorinated water	Colorimetric kit	Residual chlorine
Portable Toilets	Bacteria, disinfectants	N/A	Total/fecal coliform
Concrete & Masonry	Acid wash	pH meter	pH
	Curing compounds	pH meter	pH, alkalinity, VOCs
	Concrete rinse water	pH meter	pH
Painting	Resins	N/A	SVOCs
	Thinners	N/A	VOCs
	Paint strippers	N/A	VOCs
	Solvents	N/A	VOCs
	Adhesives	Phenols kit	Phenols, SVOCs
	Sealants	N/A	SVOCs
Cleaning	Detergents	Colorimetric kit	Surfactants, phosphates
	Bleaches	Colorimetric kit	Residual chlorine
	Solvents	N/A	VOCs
Landscaping	Pesticides/herbicides	N/A	Check with analytical laboratory
	Fertilizers	N/A	Nitrate, ammonia, phosphorous
	Lime and gypsum	pH meter	Acidity/alkalinity
	Aluminum sulfate, sulfur	pH meter	TDS, alkalinity
Treated Wood	Copper, arsenic, selenium	Metals test kits may be available	Metals
Soil Amendments & Dust Control	Lime, gypsum	pH meter	pH
	Plant gums	N/A	BOD
	Magnesium chloride	N/A	Alkalinity, TDS
	Calcium chloride	N/A	Alkalinity, TDS
	Natural brines	N/A	Alkalinity, TDS
	Lignosulfonates	N/A	Alkalinity, TDS

Figure 7-7. Decision Diagram to Determine When Construction Storm Water Sampling is Necessary



7.7.6. Collection of Construction Storm Water Samples

The conditions for collection of storm water samples from construction sites are similar to those outlined in Section 7.3.2 for industrial sites. For instance, characteristics of the storm event must be appropriate, e.g., accumulation depth, representative storm, etc., and the sample collection methods must adequately characterize the discharge. On-site environmental personnel responsible for storm water management at DoD construction sites should consult their CGP or the local permitting authority to determine specific sampling requirements.

The collection and handling of storm water runoff samples requires special care to ensure the integrity and validity of the samples. A COC form must follow the sample from the collection through the analysis process to document appropriate control of the sample prior to analysis. Additional documentation to track other information of interest, (e.g., field conditions or required field measurements) should be recorded on a field tracking form. Every sample must be collected with care to ensure that the sample is representative of the runoff being tested, collected in the right kind of container, preserved by chemical additives, and kept cold in accordance with the test method’s specifications, until delivered to an analytical laboratory. Some types of samples have very short holding times and must be analyzed before this holding time is exceeded. Sample handling requirements and documentation form the basis of your sampling QA program. More details regarding QA planning are provided in Chapter 2 of this handbook.

Before starting any sampling program, contact the analytical laboratory that will analyze the storm water samples. Make sure to select a laboratory that will provide the technical support that is needed, such as properly cleaned and preserved sampling containers and COC forms. Some laboratories can assist in identifying courier services available to transport samples to the laboratory. All these details need to be planned in advance of sample collection. The analytical laboratory should also be consulted on requirements for additional samples to be collected for QA/QC purposes.

Both field and/or analytical analysis methods can be used to meet the permit requirements. Field techniques have the advantage of providing immediate results. However, there are only a limited number of analyses that can be conducted in the field. Some constituents (e.g., pH) can be evaluated in the field with special equipment. Field samples must be collected and analyzed according to the specifications of the manufacturer of the sampling devices employed. Field equipment must be used by trained staff, and the equipment must be calibrated and maintained according to the manufacturer’s specifications.

7.7.7. Coupling Visual Observations with Sampling Results

If visual inspection of non-structural and structural storm water BMPs used to contain non-visible pollutants at a construction site indicates that a BMP has failed or been compromised, then field monitoring of the storm water for non-visible pollutants is required. Of course, any structural BMP that has been visually inspected and found breached or compromised should be immediately repaired or replaced. The intent of conducting field monitoring for non-visible pollutants is to obtain an immediate indication if storm water that is discharging from a site has been contaminated. An immediate indication of a polluted discharge requires an immediate response in the form of back tracking from the point of discharge to find the source and take appropriate measures to prevent a recurrence of a polluted discharge. If at all feasible, the contaminated discharge should be contained and prevented from being discharged off site. After taking steps to correct the failed non-structural or structural BMP, it is advisable that field monitoring be conducted to verify that pollutants are no longer in the storm water.

7.7.8. Retention of Data

Results of field measurements and laboratory analyses must be kept in the SWPPP, which is required to be kept on the project site until the Notice of Termination (NOT) is filed and approved by the appropriate control authority. It is also recommended that field training logs, COC forms, and other documentation relating to sampling and analysis be kept with the project's SWPPP. The GP requires that records of all inspections, compliance certifications, and noncompliance reporting must be retained for a period of at least 3 years from the date generated or after project completion, whichever is greater . Also note that continual monitoring of the non-structural and structural BMPs is required until the NOT has been filed and approved.

7.8. Storm Water Sampling Equipment

Automated storm water sampling and flow measurement equipment is available for a variety of situations. This equipment is designed to operate with little or no operator attention, eliminating the expense of mobilizing sampling teams during storm events. Site environmental personnel tasked with storm water sampling are encouraged to discuss their specific site conditions with equipment vendors to be certain the correct equipment is purchased.

7.9. Hazards and Safety Pre-Warnings

A project-specific HASP should be prepared for each planned storm water sampling event. Section 2.4 discusses preparation of project-specific HASPs for all types of planned sampling activities. Since storm water sampling is conducted during poor weather conditions, the following specific health and safety issues related to storm water sampling need to be considered:

- Hazardous weather conditions (e.g., wind, lightning, flooding, etc.)
- Sampling in confined spaces such as manholes
- Hazards associated with chemicals
- Biological hazards such as rodents and snakes
- Physical hazards (e.g., traffic, falling objects, sharp edges, slippery footing, and the potential for lifting injuries from opening or removing access panels and manhole covers, etc.).

It is essential that sampling personnel be aware of these hazards. Sampling personnel should be trained to evaluate potentially hazardous situations and develop ways for handling them. Since sampling hazards can be life threatening, safety must be the highest priority for all storm water sampling personnel.

7.10. QA/QC

If the permit does not contain adequate sampling information, a SAP should be prepared for collection of storm water samples at DoD industrial facilities and construction sites. Chapter 2 discusses development of SAPs. In general, the storm water SAP will document the planned activities for storm water data collection and will provide a project-specific blueprint for obtaining the storm water samples needed to verify compliance with the permit conditions. In general, the SAP will document the following:

- What samples are desired
- How, where, and when the samples are to be collected
- Required sample size, containers, and preservation
- Required sampling documentation, distribution, and storage
- The turnaround times as required by the permit or regulation
- Where samples are to be sent for analysis
- Analytical QA/QC samples
- Sample disposal.

The SAP provides an in depth description of the policies, organization, sampling activities, and QC procedures necessary for achieving project objectives. QC procedures are described in detail to ensure a high level of quality in the collection and analysis of the data and the handling of samples. Major elements in the SAP include:

- Title and approval page
- Distribution list
- Project organization
- Project overview and schedule
- Project quality objectives and measurement performance criteria
- Sample collection, documentation handling, tracking and custody procedures
- QC samples
- Analytical requirements and procedures
- Data management tasks
- Assessment and response tasks.

The SAP should be prepared well in advance of the planned sampling activities to allow for approvals, laboratory coordination, and sampling personnel training and awareness.

7.11. Sampling Equipment List

Section 3.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to storm water sampling:

- Map of sampling location(s)
- Sampling SOP
- FLB/FN
- Pen(s)
- Containers
- Preservatives
- Labels
- Markers
- Coolers
- Ice
- Packing Material
- Packaging Tape
- COC form
- Custody seals (if required)
- Decontamination storage containers, equipment, and materials
- Personal safety equipment, safety test equipment
- Field screening or testing equipment, standards, reagents, and SOP
- Testing Field Forms or Logbooks.

Chapter 8. Groundwater Sampling

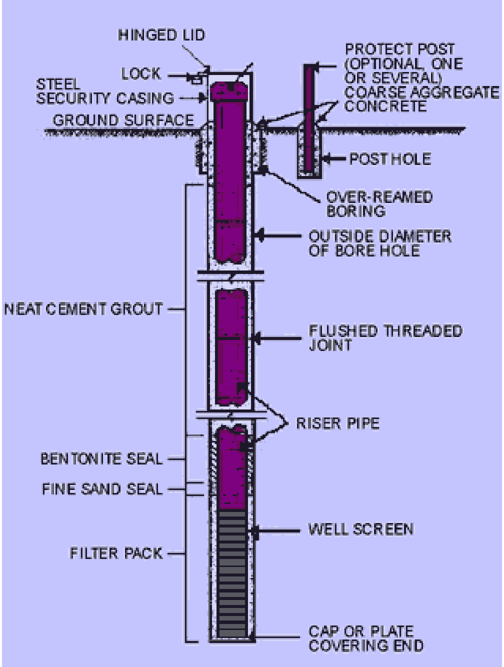
8.1. Purpose

This chapter provides procedures for obtaining representative samples of groundwater.

8.2. Scope

Groundwater monitoring wells, underground injection wells, and industrial wells are the potential sources of groundwater samples. This chapter includes the minimum criteria to be followed to obtain representative samples. Variations from these criteria should be necessary only when required by regulatory practices (e.g., state-specific requirements) or site historical data gathering practices. Analytical data derived from samples obtained in a way that does not follow the documented sampling plan should not be accepted. For construction and design of groundwater monitoring wells, refer to ASTM D5092-04, *Standard Practice for Design and Installation of Groundwater Monitoring Wells*. Figure 8-1 displays a diagram of a typical groundwater monitoring well.

Figure 8-1. Diagram of a Typical Monitoring Well



Source: Natural Resources Conservation Service.

8.3. Project Planning

This section provides an overview of the planning process for conducting a groundwater sampling program. Included in this section is a discussion of sampling strategy, development of a site-specific SAP, and QA considerations.

Before groundwater sampling begins, clear and concise objectives must be developed regarding the intent of the sampling. The objectives provide the framework for developing a sampling strategy for the site and preparing the SAP. Some common objectives of groundwater sampling might include verifying that pollutants are not migrating off site, defining the vertical and horizontal extent of pollutant concentrations from a known source, or evaluating the progress of groundwater remedial activities. Once the objectives are established, a sampling strategy can be developed.

8.3.1. Sampling Strategy

The sampling strategy is designed around the objectives and should utilize the conceptual site model. The model takes into consideration historical site use, known or suspected pollutant releases, and site geology and hydrogeology. The conceptual site model is dynamic and is constantly revised as new information is collected and processed. The model focuses on contaminant fate and transport processes, the control of geologic materials on the contaminant pathways (e.g., depositional environments, geologic structure, soil characteristics), the types of contaminants present (hydrophobic versus hydrophilic), and the processes that influence the concentrations of contaminants present such as dilution, biodegradation, and dispersion. More information on conceptual site models can be found in Chapter 2 of this handbook.

8.3.2. Site-Specific SAP

Groundwater sampling should collect a representative sample of the current groundwater conditions over a known or specified volume of aquifer. Procedures that minimize disturbance to the aquifer will yield the most representative groundwater samples. The development of a site-specific SAP is the first step toward collecting a representative groundwater sample. Each SAP is an instruction manual for field personnel and should be built around the objectives and sampling strategy. See Chapter 2 for a detailed discussion of SAP development applicable to all sampling programs. The discussion below focuses on SAP development for groundwater sampling programs specifically.

The SAP should consider a variety of factors, including site hydrogeology, the condition and accessibility of monitoring wells, suspected pollutants, DQOs, analytical methods and detection limits, the groundwater collection method, sample handling procedures (e.g., filtration, preservation requirements, COC), and safety.

The SAP cannot be implemented effectively if field personnel do not understand its contents. Many times the primary reason that groundwater sampling events are problematic is that the person preparing the SAP is not the person in the field. One method that alleviates this problem is assigning a Sampling Team Leader who is responsible for all activities in the field. The Sampling Team Leader will work closely with those individuals preparing the SAP to understand fully the objectives of the project. The Sampling Team Leader is responsible for all individuals in the field. If required, he or she also is responsible for providing additional training to field personnel before fieldwork begins.

8.3.3. QA

Groundwater monitoring programs encompass a variety of information sources and include both primary and secondary data collection. These data sources are used to continuously update the conceptual site model and allow Site Managers to make decisions regarding further investigation, additional monitoring, remediation alternatives, or site closure.

The level of data quality for each groundwater monitoring program, and many times for each specific sampling event, depends on the intended use of the data. For example, the level of QA/QC needed for groundwater samples that are collected on a monthly basis to monitor the progress of remedial activities may be different from that for groundwater samples collected because of an enforcement action. It is important to remember, regardless of the level of QA/QC for any groundwater sampling, that sample integrity be maintained during sample collection. Laboratory analysis, no matter how sophisticated, is representative only if the sample supplied to the analyst has retained its integrity. During ongoing groundwater monitoring studies, consistency of sampling is crucial to the interpretation of the data over the year or years.

To determine the level of quality required for each groundwater sampling event, Site Managers and those responsible for data integrity should begin with a systematic planning process that helps define the DQOs. The DQOs clarify the study objective, define the most appropriate type of data to collect, determine the most appropriate conditions for data collection, and specify tolerable error limits on decisions that will be used as the basis for establishing the quantity and quality of data needed to support the decision. Detailed information on QA/QC, including the systematic planning process, development of DQOs, and preparation of SAPs, is available in Chapter 2 of this handbook.

QA/QC related specifically to groundwater monitoring programs can be categorized into the following four areas:

1. Use of secondary data for preparation of the conceptual site model
2. Impacts of sampling equipment and well construction materials on sample integrity
3. Equipment decontamination, and
4. Collection of QC samples.

8.3.3.1. Secondary Data

Secondary data are those data collected in addition to sampling data. For a groundwater sampling program, secondary data may include items such as historical records regarding site use, spills, and corrective actions on neighboring properties; current and historical subsurface utility maps; locations of previous above-ground and below-ground structures; possible disposal areas; and areas containing fill materials or other antidotal agents. These data aid the site manager in selecting groundwater sampling locations or determining possible pollutants.

For secondary data, acceptance criteria are used in place of the measurement performance criteria typically used for laboratory data. In general, acceptance criteria are used to assess secondary data adequacy and evaluate uncertainty in the results derived from the use of secondary data sources. For example, the Project Manager for the groundwater monitoring program may require interviews of former employees to validate the location of disposal areas found on historical site maps. In this case, the acceptance criteria are the verbal confirmation of the disposal areas by the former employees. The SAP for each groundwater monitoring project will explain the acceptance criteria for determining which sources of data are sufficient to support the project objectives.

8.3.3.2. Equipment Specifications for QC

The materials used for constructing groundwater sampling equipment are critical to the collection of valid monitoring data, particularly when volatile organic, pH-sensitive, or valence-reduced chemical constituents are being evaluated. The construction materials that come into contact with the sample are as critical as the composition of the laboratory sample containers. The recommended materials for bailers, pump parts, tubing, other sampling devices, and associated apparatuses in decreasing order of preference are as follows: Teflon®, stainless steel 316, stainless steel 304, polypropylene, linear polyethylene, polyvinyl chloride (PVC), Viton®, and conventional polyethylene.

Most regulatory programs require that bailers be constructed of Teflon® or stainless steel. Additionally, any other devices contacting the water to be sampled should be constructed of Teflon® or stainless steel. Exceptions to this requirement should be confirmed and approved by the regulatory program having project oversight authority. Tubing used in well evacuation may consist of materials other than Teflon®, but this tubing may not be used for sample collection. Tubing should be dedicated for use in each individual well for that particular sampling event.

8.3.3.3. Equipment Decontamination

Decontamination of existing and new equipment is required prior to use in the field. Section 8.5.3 describes decontamination procedures for various types of groundwater sampling equipment. These procedures must be followed to prevent cross-contamination between sites and within the site. Equipment field blanks may be collected at the start and end of the sampling event to determine the cleanliness of the sampling devices and evaluate the cleaning techniques used in the field. Decontamination procedures for all equipment that contacts groundwater must be included in the SAP.

8.3.3.4. QC Samples

In an attempt to identify external variables affecting groundwater sample integrity, a program of QC blanks should be initiated. For volatile parameters, the QC blank sample program is a two-track approach using both a trip and a field blank. The trip blank acts as a check on potential contamination sources in the sample container, method blank water (including preservative), and sample transport and storage. The field blank acts as a check on the cleanliness of the sampling equipment, potential atmospheric contamination, and the effects of sampling procedures such as preservation on the analytes of interest. Complete documentation on the sources of these materials will assist with any problem solving.

Equipment field blanks may be collected at the start and end of the sampling event to determine the cleanliness of the sampling devices and evaluate the cleaning techniques used in the field.

Trip blanks, field blanks, and rinsate blanks typically are prepared with metal-free and organic-free water purchased from a chemical supply company or provided by the laboratory that is performing chemical analyses on the groundwater samples.

Field duplicates or splits are collected in the field in double the number of bottles required for the regular sample. A duplicate sample is collected concurrently, under comparable conditions, with a first sample. Duplicate samples are QC samples that are used to assess data repeatability based on field conditions. Split samples are two or more representative portions taken from one sample in the field or in the laboratory and analyzed by different analysts or laboratories. Split samples are QC samples that are used to assess analytical variability and comparability. Samples sent to the same laboratory are duplicates, and samples sent to a different laboratory are splits. Both duplicate samples and split samples shall be field homogenized and shipped to the laboratories as blind samples. Duplicate samples provide a total precision of field sampling precision and lab analysis. Analytical data from these samples will determine field precision (duplicate) or project precision (split).

Field spikes are prepared in compliance with permit or regulatory requirements. Field spikes determine field accuracy or laboratory accuracy. To prepare a field spike, a known amount of contaminant is placed or spiked into the sample in the field. Samples and spikes are handled in the same manner. Field spikes help to assess analytical method performance and contaminant deterioration or degradation during sample handling, transport, and analysis.

8.4. Hazards and Safety Precautions

The HASP should address all anticipated hazards for each task. The following discussion contains common (and commonly overlooked) hazards associated with groundwater sampling activities. Refer to Chapter 2 for specific hazard identification techniques and detailed control measures.

8.4.1. General Safety

Some of the general safety hazards associated with groundwater sampling programs include the following:

- Moving sampling equipment and containers across rough terrain either by hand or by driving off road
- Conflicts with other uses of the area, such as traffic conflicts when sampling next to roads
- Sampling from deep sampling monitoring wells or open holes using hand-held sampling equipment.

8.4.2. Subsurface Hazards

Groundwater sampling, particularly monitoring well installation, is an intrusive activity that could expose sampling personnel to unidentified subsurface hazards, as discussed below.

8.4.2.1. Buried Munitions

If the probability of encountering buried munitions during the sampling program is moderate to high, a RESS waiver from the relevant explosives safety authority may be required prior to disturbing the site. In addition, a qualified explosives safety expert will be required to accompany the investigation team. See DoD TP18 for the minimum qualifications for unexploded ordnance technicians and personnel. The site should be checked by qualified personnel with ground-penetrating radar and/or metal detectors before any samples are collected. Only trained and certified personnel should handle or sample explosive or suspected explosive materials.

8.4.2.2. Buried Utilities

The use of in situ groundwater sampling probes to install monitoring wells can damage buried utilities, including electrical and telephone lines and gas, steam, water, and industrial pipelines. Facility records should be reviewed carefully to identify any buried utilities in the sampling area. The site should be checked with ground-penetrating radar and/or metal detectors if there is any question as to the presence of subsurface utilities.

8.4.2.3. Toxic Chemicals

Collecting groundwater samples poses the risk of exposure to hazardous chemicals. The following major exposure scenarios should be evaluated.

- **Skin Contact with Contaminated Water.** Taking groundwater samples using hand-held samplers usually results in the requirement to handle wet equipment that can drip and splash.

Decontamination of sampling equipment also can result in splashes of contaminated water. Field sampling and decontamination personnel should wear waterproof rain gear or coated Tyvek® coveralls if there is a possibility of contact with contaminated water.

- **Ingestion of Contaminated Water.** Ingestion can occur when food or drink is exposed to contaminated water or is picked up with contaminated gloves. Field personnel should not eat, smoke, or chew gum or tobacco when working with contaminated water. Drinking water should be kept in closed squirt bottles or should be stored outside the contaminated area.
- **Inhalation of Toxic Vapors**. The exposure of contaminated water to ambient air may generate toxic vapors from the evaporation of VOCs. The HASP should establish air-monitoring requirements for intrusive work and specify what air monitoring equipment be used and the criteria for the use of respirators. Either air-purifying respirators or supplied-air respirators may be required, depending on the toxicity of the chemicals present, the capabilities of the monitoring equipment, and the warning properties of the vapors.
- **Confined Space Hazards.** In general, wells more than 4 feet deep are considered confined spaces. However, any well installed in contaminated soil or water should be considered a confined space, because flammable vapors or toxic gases, including hydrogen cyanide and hydrogen sulfide, could accumulate. All applicable confined space entry requirements should be imposed and detailed in the HASP.

WARNING: Care should be taken to avoid breathing in vapors that may escape from the well.

8.5. Field Preparation

The key to any successful field sampling program is preparation. This section describes the preparations that should be made prior to personnel entering the field to conduct a groundwater sampling event.

8.5.1. Access and Identification

Access to monitoring wells may be difficult, and the wells themselves hard to locate in the field. The Field Team Leader responsible for the groundwater sampling should obtain recent well location maps prior to the sampling event. In addition, because access to some locations may require permission from current owners or operators of the site, the Field Team Leader should contact the site owner or operator in advance of each sampling event to obtain access and inform those individuals responsible for the site that groundwater sampling is scheduled. Monitoring wells usually have a friction cap or a screw cap and should be locked. Therefore, keys to unlock the wells and tools for removing caps often are necessary.

If several monitoring wells must be sampled, proper identification of each well is essential. The well permit number or any other assigned number should be known. If numbers are not assigned, a precise field description of each well location will avoid confusion of the sample results. When several monitoring wells of known or suspected contamination will be sampled, the least-contaminated well should be sampled first. Subsequent samples then should be collected from wells in an ascending order of contamination. Well head readings using PIDs or FIDs can aid in determining the order in which wells should be sampled by providing information on contamination levels.

8.5.2. Sampling Equipment Selection

The equipment used for specific groundwater sampling events can vary greatly, depending on the following factors:

- Type of well
- Depth of well
- Diameter of well casing
- Depth to water
- Contaminants likely to be encountered
- Analytes of interest
- Length of open hole (bedrock well)
- Type, slot size, and length of screen

- Expected recharge rate of well.

The equipment required for groundwater sampling generally falls into two categories: 1) equipment used to evacuate water in the well casing, i.e., well development, and 2) equipment used to collect a discrete sample for analysis. However, in some instances, the device used for evacuation may be the same as that used for sample collection.

8.5.2.1. Field Measurement Equipment

Prior to well evacuation, groundwater level measurements are made in each groundwater monitoring well. If either floating or settled organic phases are expected in the groundwater, the thickness of these phases also should be measured. The equipment typically used to measure groundwater levels includes the following:

- Electronic tape that signals to the user when the water level is reached
- A hydrocarbon interface probe affixed to an electronic tape to measure the depth to either the floating or the settled organic phase, and the thickness of the phase.

8.5.2.2. Well Evacuation Equipment

Evacuation or purging of the water column in a monitoring well is required prior to sample collection to remove the standing water column and induce groundwater flow from the surrounding formation into the well. One exception to this standard procedure is if the objective of the sampling event is to determine the presence of dense or light non-aqueous phase liquids or stagnant water. The following types of pumps are available for well purging:

- Suction lift pump/centrifugal pump
- Portable submersible pump
- Peristaltic pump
- Air lift pump
- Bladder pump (gas squeeze pump)
- Packer pump
- Gas piston pump
- Gas displacement pump
- Inertial pump.

8.5.2.3. Sample Collection Equipment

The types of equipment available for groundwater sample collection include the following:

- Bottom fill bailer (single or double check valve)
- Peristaltic pump
- Bladder pump
- Packer pump
- Inertial pump
- Syringe sampler
- Disposable equipment.

Site-specific sampling conditions will dictate the optimal sampling equipment. Generally, sampling equipment that minimizes agitation, air content, gas exchange, and depressurization is preferred.

A list of additional sample collection equipment is provided at the end of this chapter.

8.5.3. Equipment Cleaning and Decontamination

For each sampling event, all field measurement and sampling equipment that will enter the well must be cleaned prior to its entry. Field measurement equipment, such as water level indicators, should be cleaned in the following manner:

1. Wipe with a paper towel to remove visual debris
2. Wash with tap water and a laboratory-grade glassware detergent
3. Rinse with tap water
4. Perform an ASTM Type II water rinse.

Sampling equipment should be cleaned using documented cleaning procedures. The sampling equipment then should be wrapped in clean foil and dedicated to a specific well for the day’s sampling. The sampling equipment should remain wrapped in this manner until immediately prior to use. Additionally, bailers and sample bottles must be physically separated from pumps and generators during transport and storage. Pumps and equipment not amenable to precleaning should be field-cleaned using documented cleaning procedures. Down-hole devices such as water level indicators should not be transported in a vehicle storing gasoline or gasoline powered equipment or other volatile contaminants such as degreasers, cleaning solvents, and other volatile organics.

8.6. Sampling Monitoring Wells

This section describes the procedures for sampling monitoring wells. It describes collection of field measurements, well evacuation procedures, and groundwater sampling procedures.

8.6.1. Field Measurements

CAUTION: Be certain that the proper well is being selected. The misidentification of a sampling point in the field will result in data that may adversely affect important decisions.

8.6.1.1. Physical Measurements

Once a well has been located and properly identified, note the field measurements listed below in the FLB/FN.

- Diameter of protective outer casing
- Security and integrity of the well
- Well number and well permit number
- Inner diameter and construction material of inner well casing
- Total depth of well from the top of the inner casing or surveyor's mark, if present (measured to 0.01 foot, or as appropriate).

8.6.1.2. Water Level Measurements

Well depths and water table depths can be determined using various measuring devices. A commonly used device is the electronic water level indicator. This unit has a tape divided into incremental measurements of 0.01 feet and two conductors forming a probe. When groundwater is encountered, the circuit is completed causing a signal (e.g., light, meter, or audible buzzer) to activate. The depth to groundwater is then measured from this point to the reference mark on the inner casing of the monitoring well.

Water indicator paste or gel acts as a colorimetric test method when the paste comes into contact with water. It is applied to the bottom few feet of a measuring tape or rod. The tape or rod is then lowered into the well and remains for less than 1 minute. The wetted tape/stick gives the depth to the top of the liquid and the color change section indicates the depth to water. This procedure is accurate to ± 0.02 feet.

Wells with a non-aqueous phase liquid layer on the surface pose a problem when measuring the level of groundwater. A more accurate and easier device to use is the interface probe. This probe uses an optical sensor to determine if the probe is in liquid, and a conductivity sensor to determine if the probe is in water. When using this probe, each phase can be measured independently. The hydrocarbon/air interface reading should be taken first, going down from the air to the hydrocarbon surface to prevent any dripping hydrocarbons from enhancing the thickness reading. The hydrocarbon/water reading is best taken going up from the water to the hydrocarbon layer to prevent hydrocarbons from coating the conductivity probe, which also would enhance the hydrocarbon thickness reading. Prior to taking the hydrocarbon/water reading, the probe should be lowered quickly through the hydrocarbon layer, minimizing the contact time of the probe in the hydrocarbon phase.

The key to accurate readings by any method is proper collection of measurements from the same survey point, preferably by the same person and tape to avoid any procedural differences. Each reading should be made three to four times. All well measurements should be performed the same day and prior to evacuation of any wells that could influence groundwater elevations in the area of investigation.

Water level elevation equipment should be properly decontaminated to avoid cross-contamination. In certain circumstances, sensitive components of an interface probe may be compromised by the use of standard decontamination solvents. The manufacturer's specifications for decontaminating interface probes should be consulted before using any solvent.

For each water level measurement, the following data should be recorded in the FLB/FN:

- Depth from casing top to water (recorded to 0.01 foot, or as appropriate)
- Thickness of floating product, if any
- Calculation of the linear feet of water in well by subtracting the depth to water from the total depth of well.

Note: Water levels should be obtained from all wells prior to purging and sampling the first well, thus avoiding interference problems. This procedure also allows personnel to determine if any well is damaged or may pose a problem for sampling.

Using the physical measurements and water level measurements, the amount of water within the entire well casing can be calculated by multiplying the linear feet of water by the volume per foot for the proper diameter casing.

Table 8-1. Volume of Water per Linear Foot in Various Casing Sizes

Casing Diameter (foot)	Gallons/Linear foot
2 inch (0.1667)	0.1632
4 inch (0.3333)	0.6528
6 inch (0.5000)	1.4688
8 inch (0.6667)	2.6112
10 inch (0.8333)	4.0800
12 inch (1.0000)	5.8752
Example:	
Total depth of well casing:	100 ft.
Depth to water:	-20 ft.
Linear feet of water:	80 ft.
2-inch casing:	x 0.1632
Amount of water in casing:	13 gal.

Table 8-1 above shows the volume of water per linear foot of various casing sizes (inner diameter) of monitoring wells.

The amount of water in the casing then should be multiplied by three to determine the minimum volume to be purged from the well prior to sample collection. The total volume purged should not exceed five times the amount of standing water in the well.

Alternatively, the following formula can be used to determine the number of gallons in any diameter well:

$$\text{Number of gallons} = 5.8752 \times C^2 \times H$$

where: C = casing diameter in feet
H = height of water column in feet

8.6.1.3. Physio-Chemical Parameters

Information including specific conductance, pH, temperature, DO, oxidation-reduction potential, and turbidity may be recorded during well purging and before and after sample collection.

In some cases, additional physio-chemical analyses may be required to support the engineering of future or current treatment processes. Samples for these purposes are often collected for analysis of common cations (Na^+, K^+, Ca^{+2}, and Mg^{+2}) and anions (CO_3^{-2}, $SO_4^{-}2$, Cl^-, and NO_3^-), dissolved iron and manganese, TDS, TSS, TOC, and dissolved carbon. These are common and inexpensive analyses.

8.6.2. Well Development and Purging

Well development and well purging are two similar operations that are conducted to ensure that groundwater from a surrounding formation flows into the monitoring well for sampling. Each of these operations is discussed in the following subsections.

8.6.2.1. Well Development

Well development typically is performed shortly after construction of the monitoring well to ensure that free-flowing groundwater will enter the monitoring well. Well development also is designed to provide removal of the following:

- Drilling fluid residues remaining in the bore hole or surrounding aquifer
- Imported drilling water lost to the aquifer during the drilling procedure
- Groundwater in the bore hole or surrounding aquifer that has been affected by the drilling process or drilling or well construction materials.

As well as:

- Restoration of the hydraulic properties of the formation immediately surrounding the monitoring well.

The length of time for groundwater conditions to become representative (stabilization period) at and near the monitoring well will vary depending on site hydrogeologic conditions, drilling methods, and monitoring well development methods. Groundwater flow velocities are typically less than 1 foot per day, and natural flushing rates are generally slow. If a monitoring well is drilled, installed, and developed so that a 14-foot radius around the well was left as unrepresentative, and a natural groundwater flow rate was 1 foot per day, it would take 14 days for representative groundwater to reach the well. Sampling a monitoring well immediately after development generally will not be representative of the static groundwater quality conditions at the horizontal and vertical location of the monitoring well intake interval. Therefore, all newly constructed and developed monitoring wells should be allowed to stabilize and equalize with the aquifer for a minimum of 2 weeks prior to sampling.

The installation and construction of monitoring wells may alter the quality of groundwater in the surrounding aquifer. Site-specific subsurface conditions should be used to determine the appropriate well development techniques. Many times, a combination of the techniques discussed below will be necessary to produce a properly developed monitoring well. Also discussed below are certain outcomes inherent to well development techniques that can be mitigated by following the 14-day stabilization period:

- High velocity air jetting, air lift, or surge block development methods may introduce air into the aquifer surrounding the monitoring well. This air has the potential for altering groundwater quality, particularly for VOCs.
- Over-pumping of a monitoring well for development may draw groundwater to the monitoring well from considerable distances. This water may not be representative of the horizontal and vertical location of the monitoring well, especially so for isotropic and/or bedrock aquifers.
- Organic drilling fluid residues and inorganic residues of bentonite have been found to remain in and near wells, even after proper development. These residues have been found to affect water quality, including the chemical oxygen demand of groundwater samples, for up to 100 days after completion of development.
- Non-aqueous phase liquid contaminants may be pushed away or drawn to a monitoring well location during development, depending on the development method, resulting in non-representative groundwater samples.
- Suspended sediment not completely removed by development and not allowed to settle out may affect the quality of groundwater samples obtained from the well. Therefore, a period of time is required to allow a sand/gravel pack to settle around a monitoring well screen.

Groundwater pollution investigations often base expensive site-related investigatory and remedial action decisions on initial (first sampling event after development) groundwater sample analyses. Therefore, before groundwater samples are collected, a complete understanding of the design, construction, and hydrogeologic setting of the monitoring well is necessary to interpret properly any analytical results.

8.6.2.2. Well Purging

It is generally accepted that water in the well casing is non-representative of the formation water and should be purged prior to collection of groundwater samples. Monitoring well purging is a procedure that draws fresh groundwater from the surrounding formation into the well immediately prior to sample collection. Wells are purged to some extent for the following reasons:

- The presence of the air interface at the top of the water column results in an oxygen concentration gradient with depth
- Loss of volatiles up the water column
- Leaching from or sorption to the casing or filter pack

- Chemical changes due to clay seals or backfill
- Surface infiltration.

Many methods may be used for well purging. Not all are acceptable under all conditions. The selection of a method is usually dictated by the depth to water and local agency requirements. The preferred and most commonly used methods involve the use of a centrifugal or peristaltic pump (when the depth to water is < 25 feet) and a submersible pump (when the depth to water is > 25 feet).

It is important to ensure that the purging procedure or equipment does not cause cross-contamination from one well to the next. Therefore, the preferred method employs dedicated tubing (new dedicated linear polyethylene ASTM drinking water grade) and pumps. Because it may not be practical to dedicate a pump to a specific well, it is permissible to decontaminate this equipment between wells. Methods for decontamination should be specified in the site-specific SAP.

Prior to purging, check the well for floating product. During purging, the pump intake or tubing should be kept at a maximum distance of 6 feet below the water level to prevent disturbance of sediment on the bottom of the well. The pump intake or tubing should be lowered as the water level decreases to maintain this distance and ensure sufficient distance from the air/water interface. In instances where the total depth of standing water in the well casing is less than 6 feet, begin purging near the top of the water column and lower the tubing as stated above. Following this procedure, Field Sampling Personnel should ensure that all static water is removed prior to sampling.

Note: The disposal or discharge of floating product or hydrocarbons, and the discharge of highly contaminated water, may require special purge water collection and disposal procedures.

Regardless of the purging procedure used, the evacuation rate should not exceed that of well development. Such an exceedance would cause a "redevelopment" of the well, resulting in a turbid sample. Cleaned equipment entering the well should not be allowed to contact the ground or any other potentially contaminated surfaces such as gasoline pumps. If contact should occur, the item should not be placed in the well or used for evacuation.

The following information should be recorded in the FLB/FN for each monitoring well sampled:

Before Purging

- Date, time, and weather conditions
- Well number and permit number
- PID or FID reading taken from the well immediately after the cap is removed
- Presence and thickness of free product
- pH, DO, temperature, and specific conductivity
- Total depth of well from the top of inner casing or surveyors mark if present
- Depth from the top of inner casing to the top of screen
- Depth from the top of inner casing to water
- Estimated water volume in well.

After Purging

- Start and end time purging
- Purge method
- Purge rate(s)
- Total volume purged
- pH, DO, temperature, and specific conductivity.

After Sampling

- Start and end time for sampling
- pH, DO, temperature, and specific conductivity
- Sampling method.

Any field observations made during the groundwater sampling event (e.g., slow recharge, turbidity, odor, sheens, and PID or FID readings) also should be reported.

Air-sensitive parameters such as DO, pH, temperature, and specific conductance are best analyzed with the use of a flow-through cell, eliminating sample exposure to air. However, monitoring of these air-sensitive parameters for well stability may not be a reliable indicator of when to collect a representative sample. Therefore, if a constant monitor is not used during well purging, a sample should be collected within 2 hours after 3 – 5 volumes of water have been purged from the well. The volume evacuated and the evacuation rate should be recorded after each purge and sample event, and repeated for subsequent sampling events. This procedure should provide consistent samples from each well.

Every reasonable effort should be made to keep pumping rates low to avoid over-pumping or pumping the well to dryness. Pump rates may be adjusted and pumping times extended to remove the required three to five well volumes. Sampling should never occur more than 24 hours after purging. To avoid altering the hydrogeologic properties of the aquifer near the well, the evacuation rate of a monitoring well should not exceed that of the development of the well.

In some situations, evacuation of 3 – 5 volumes may be impractical in wells with slow recoveries. If a well has been pumped to near dryness at a rate of less than 0.5 gallons per minute, the well should be allowed to recover to a volume sufficient for sampling. If necessary, sampling within the 2-hour limit may be exceeded to allow the well to recover sufficiently for sampling. If a well has been pumped to dryness, a minimum of 20 minutes of waiting time is required prior to sampling, or follow regulatory requirements.

There are several reasons why the well should not be pumped below the level at which the groundwater enters the well. In certain formations, water entering the well at the top of the screened area will fall into the pumped dry well. This cascading effect may aerate the groundwater to be sampled, thus resulting in the loss of VOCs. Secondly, pumping to dryness can cause dehydration of the saturated zone; again, VOCs may be lost due to aeration within this zone. Additionally, other contaminants may absorb to formation materials where a dehydrated zone is created. As a result, samples collected upon the recharge of a well pumped to dryness may not correctly characterize groundwater quality.

Another problem with excessive purging is the entrainment of soils and other solids from the formation into the well. Soils may contain either metals or hydrophobic organic compounds that become entrained into the well, resulting in an overestimation of certain analytes. Soil particles can be filtered from the sample; however, filtration may remove potentially mobile (contaminant-associated) constituents, resulting in artificially low concentrations. One method to reduce entrainment of soil and other particulates into the well is by using low-flow purging and sampling techniques.

8.6.2.3. Low-Flow Sampling and Purging

The term “low-flow” refers to the pumping rate during purging and sampling, which affects both the velocity that is imparted to the formation pore water in the immediate vicinity of the well screen, and drawdown of the water level in the well. When performed correctly, low-flow sampling collects water from the formation surrounding the screen and avoids collecting stagnant water from any blank casing above the screen. Low-flow purging and sampling reduces the effects of the sampling on the system being sampled.

If little or no information about the groundwater system is known, the pump intake may be set at or near the middle of the well screen. However, if available, knowledge of the well-specific hydrogeology, boring logs, well construction, and well location relative to the lateral and vertical contaminant distribution in the formation should be considered when placing the pump intake. The source of water moving into the screened interval of the well under pumping conditions is strongly influenced by the hydrogeology. In wells where the screened interval intersects zones of contrasting hydraulic conductivities, the composition under steady state purging conditions will be flow-weighted in proportion to the hydraulic conductivity from any given point in the flow field.

The objective is to pump in a manner that minimizes stress (drawdown) to the system to the extent practical, while taking into account established site sampling objectives. The pumping rate must be low enough to minimize mobilization and entrainment of particulate matter that is not mobile under non-pumping conditions. Typically, flow rates between 0.1 and 0.5 liters per minute are used; however, the selection of an appropriate flow rate is dependent on site-specific hydrogeology. Water level drawdown provides the best indication of the stress imparted by a given flow rate for a given hydrological situation. Measure the initial static water level, and then monitor the water level of the well during purging using either a continuous water-level measurement device such as a pressure transducer or bubbler, or make periodic measurements (every 1 to 2 minutes) using an electric tape or similar measuring device. If drawdown is rapid and continuous, reduce the pumping rate until drawdown stabilizes. If drawdown is very slow or imperceptible, the pumping rate can be increased. When drawdown and water-quality indicator parameters (e.g., conductivity, DO) have stabilized, sampling can begin in accordance with the site's approved sampling and analysis plan.

In general, the advantages of low-flow purging and sampling include the following:

- More representative of the mobile load of contaminants (dissolved and colloid-associated)
- Minimal disturbance of the well, thereby minimizing sampling artifacts
- Less operator variability, and greater operator control
- Reduced stress on the formation (minimal drawdown)
- Less mixing of stagnant casing water with formation water
- Reduced need for filtration
- Smaller purging volume, which decreases waste disposal costs and sampling time
- Better sample consistency, and reduced artificial sample variability.

Some disadvantages of low-flow purging include the following:

- Generally not suitable for use in low-yield wells
- Higher initial capital costs for items like a variable flow-rate pump or flow-through cell
- Greater set-up and purging time in the field
- Need to transport additional equipment to and from the site
- Increased training needs.

Even in low-yield wells, low-flow purging and sampling may be more suitable than high-flow, or purging to dryness and sampling by bailer or pump as soon as the well recovers. Aeration of the water column in a well caused by excessive drawdown or pumping a well dry can significantly alter water quality, affecting redox-sensitive metals, and causing increased turbidity and loss of volatile constituents. If a well is evacuated to dryness or below the well screen, sampling records should document the event because sample integrity may be severely altered. For very low-yield wells, or wells with severe turbidity problems, passive or no-purge sampling methods may be more appropriate and should be considered.

For additional information and more specific procedures, see ASTM D 6771, *Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Groundwater Quality Investigations*.

8.6.3. Pumps

This section provides a discussion on various pumps used for sampling groundwater.

8.6.3.1. Suction Lift Pumps/Centrifugal Pumps

Suction lift pumps (e.g., diaphragm and centrifugal pumps) are used at the ground surface with polyethylene tubing inserted into the well. They are used to evacuate the well prior to sampling. The tubing should be new, dedicated to a particular monitoring well, and equipped with a decontaminated foot check valve to avoid having aerated water from the pump spill back into the well.

If a foot check valve is not used, care should be taken to ensure that the entire pump impeller chamber is drained after use and then thoroughly decontaminated. Also, when removing tubing without a foot check valve after evacuation, the pump should continue to operate to keep purged water remaining in the tubing and pump chamber from spilling back into the well.

The limitation posed by this type of pump is its suction capability. Generally, the water level must be within 25 feet of the ground surface.

Note: Take care to ensure that *only* ASTM Type II or better quality water that is free from chlorine residual and potential contaminants is used to prime the centrifugal pump. These pumps may be used only for well evacuation, not for groundwater sampling.

8.6.3.2. Portable Submersible Pumps

When the depth to water is greater than 25 feet, and if the diameter of the well casing will allow, a portable submersible pump should be used. The pump should be lowered carefully into the well, trailing a discharge hose, electrical cables, and a security cable constructed of approved material (e.g., single-strand stainless steel or polyethylene). The security cable should support all of the weight of the pump. Hoses and cables can be bundled together at 10-foot intervals with plastic electrician's ties or stainless steel clamps.

CAUTION: Duct or electrical tape must not be used at a level that will be submerged into the water column. It is important that the hose and electrical line be fed so they do not jam between the pump and the casing. Similarly, the hose and electrical line must be pulled up ahead of the pump during removal.

Once the end of the purged line is fitted with a gate valve in the closed position, the submersible pump should be lowered to the appropriate depth. The pump then can be turned on and the gate valve adjusted to provide the correct flow rate. During evacuation, it may be necessary to lower the pump as the static groundwater level drops.

If a portable gasoline generator is used, it should be placed downwind and at some distance away from the well so fumes from the generator will not affect sample quality. The generator should only be operated during the purging process and not during sample collection.

The pump should be fitted with dedicated tubing for the discharge of evacuation water. As with suction lift pumps, submersible pumps should be equipped with a check valve to avoid having water from the pump spill back into the well. If the same submersible pump is to be used for more than one well, then the pump should be decontaminated between well locations to ensure that no cross-contamination occurs.

Submersible pumps are susceptible to clogging. Turbid groundwater or poorly developed monitoring wells are likely to impede the evacuation process. Take care not to let the pump draw from the bottom of the well where silts and sands may be taken up by the pump.

8.6.3.3. Peristaltic Pumps

A peristaltic pump is a self-priming suction lift pump used at the ground surface. It consists of a rotor with ball bearing rollers. One end of dedicated tubing is inserted into the well. The other end is attached to a flexible tube that has been threaded around the rotor, out of the pump, and connected to a discharge tube. The liquid moves entirely within the sample tube, with no part of the pump contacting the liquid. The bottom length of tubing should be equipped with a foot check valve to avoid having water from the pump and tubing spill back into the well.

Use of a peristaltic pump for well evacuation is limited to its suction capabilities. Generally, a peristaltic pump cannot be used to evacuate wells with a depth to water of greater than 25 feet. Due to the volume present in large-diameter and high-yield wells, peristaltic pumps are not recommended.

8.6.3.4. Air Lift Pumps

Air lift pumps generally are used for well development and not for well evacuation prior to sampling. If logistics dictate that air lift pumps are the only alternative for evacuation, they should be used only for wells screened below the water table. Tubing connected to air lift pumps should be placed above the well screen because air may become trapped in the screen or filter pack. Entrapped air can alter the oxidation-reduction potential of the aquifer material around the well bore, which can affect the chemical composition of groundwater samples. In addition, only oil-free compressors should be used.

8.6.3.5. Bladder Pumps (Gas Squeeze Pumps)

A bladder pump consists of a stainless steel cylindrical housing that encloses a flexible membrane. A screen is attached to filter any material that can clog the check valves located above and below the bladder. The pump works as follows: water enters the membrane through the lower check valve, and compressed gas is injected through a separate line to the space between the bladder and the pump housing. As the bladder is squeezed, the water in the bladder closes the lower check valve and flows through the upper check valve. As the air pressure is released, the upper check valve closes and water enters the pump through the lower check valve. The compressed gas does not come into contact with the sample water.

The bladder pump is used much like a portable submersible pump, except that no electrical lines are lowered into the well. The source of gas for the bladder is either bottled gas or an on-site oil-less air compressor. Disadvantages posed by using the bladder pump include the large gas volumes needed, especially for greater depths, and the potential for bladder rupture and slow evacuation rates.

The preferred material of construction for bladder pumps and any tube, joint, or other fixture that remains in contact with the groundwater is Teflon® or stainless steel.

8.6.3.6. Packer Pumps

Packer pumps consist of two expandable bladders that, when inflated, isolate a section of the well bore between them. They deflate for vertical movement within the well. The advantage of this type of pump is that a smaller volume of water is required to be evacuated prior to sampling. Also, several zones within a single well can be sampled. The sampler must be sure the zone that is being sampled and packed is isolated from the other zones.

Packer pumps are constructed of rubber and can be used with submersible, gas lift, and suction pumps. Exposures to high-level contamination may deteriorate the rubber with time.

8.6.3.7. Gas Piston Pumps

The gas piston pump provides continuous sample withdrawal at depths greater than possible with most other methods. The pump consists of a stainless steel alternating chamber between two pistons. Pressurization of the alternating chamber activates the pistons, which allows water to enter during the suction stroke, and forces water to the surface during the pressure stroke.

8.6.3.8. Gas Displacement Pumps

Gas displacement pumps work by using gas to force water out of a discharge line. They consist of a cylinder with a check valve and two lines, an air supply line and a water discharge line, and a connection to the top. As the pump is lowered into the water, it fills by hydrostatic pressure. When air pressure is applied, the check valve seals and water is forced out of the discharge line. When the air pressure is released, the pump chamber fills and the cycle repeats.

8.6.3.9. Inertial Pumps

The inertial pump consists of a single tube or pipe with a foot check valve at one end. The check valve allows water to enter the pipe but stops it from draining out.

The pump is operated by raising and lowering the tube over a short distance with rapid strokes. The movement causes the water inside the pump to travel upward due to its inertia. The movement can be accomplished manually or with a powered unit.

The advantages of using an inertial pump are its ease of operation and low cost. However, the inertial pump has several disadvantages, which are listed below.

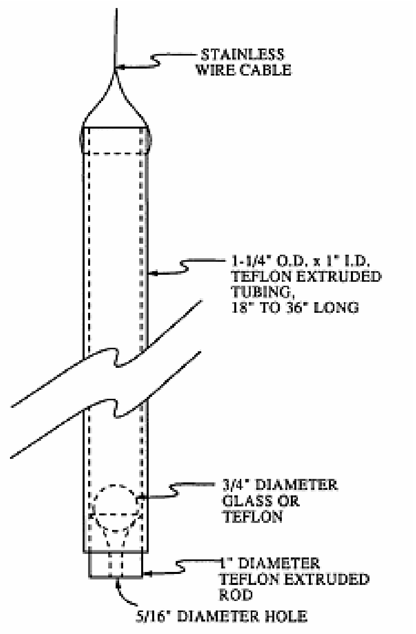
- Its manual operation is labor intensive, although mechanical advantage devices are available
- The tubing and foot assembly must be dedicated to a well
- Use in slow-recharge wells may cause the water level to drop significantly and result in

aeration of the water column during purging of the well. Conversely, the inertial pump device can be overwhelmed in a rapidly recharging well leading to insufficient evacuation of the water column.

8.6.3.10. Hand Bailing Techniques

Hand bailers come in a variety of sizes and volumes to accommodate most well casing diameters; see Figure 8-2 for an illustration of a bottom fill bailer. Hand bailing may be conducted if no other method of evacuation can accomplish the task and the procedure is specified in the SAP. However, bailing is the least recommended procedure for well purging due to the potential for aerating the well water or possibly introducing contaminants during the bailing procedure. Bailing is the least recommended method of purging when samples are to be collected for VOC analysis. If hand bailing is the method of evacuation, it must be performed with a laboratory-cleaned and dedicated Teflon® or stainless steel bailer. An additional laboratory-cleaned and dedicated bailer is required for sample collection.

Figure 8-2. Bottom Fill Bailer



The bailer should be lowered slowly into the well, using care not to aerate the groundwater to be sampled. The preferred apparatus is a Teflon®-coated stainless steel cable attached to a low-gear-ratio winch, which is connected to a tripod standing over the well. This apparatus is preferred because it provides the most reproducible bailing method. If this apparatus is not available, the bailer may be lowered by hand using a Teflon®-coated stainless steel leader.

Note: Braided stainless steel cable is coated with manufacturing oils that make decontamination difficult. Therefore, Teflon® coated, stainless steel is required for the bailer leader since it will come into contact with the groundwater.

Take care if using stainless steel cable clamps to secure the leader to the bailer. The integrity of the Teflon® may be compromised by compression while tightening the clamps, thus exposing the braided wire. All cut ends of leaders must have an end cap to prevent exposure of the stainless steel wire.

8.6.4. Groundwater Sampling Procedures Using Monitoring Wells

After purging the required volume of water from the well, sampling can begin. If the well is a quick recharger, sampling of the well should occur immediately after evacuation. In most cases, the time lapse between evacuation and sampling should not exceed 2 hours.

If several monitoring wells must be sampled, proper identification of each well is essential. The well permit number or any other assigned number should be known. If numbers have not been assigned, a precise field description of each well location will avoid confusion of the sample results. When several monitoring wells of known or suspected contamination will be sampled, the least-contaminated well should be sampled first. Subsequent samples then should be collected from wells in ascending order of contamination. Well head readings using PIDs or FIDs can aid in determining the order in which wells should be sampled by providing information on contamination levels.

Field personnel should pay strict attention to proper decontamination procedures during groundwater sampling; see Section 8.3.3.3.

A variety of techniques and equipment (including several of the pumps discussed in Section 8.6.3) can be used to collect groundwater samples, as discussed in the following subsections. The order in which samples should be collected from each well, regardless of sampling device, is as follows:

1. VOAs
2. Purgeable organic carbons
3. Purgeable organic halogens (POXs)
4. Total organic halogens (TOXs)
5. TOC
6. Base neutrals/acid extractables
7. Oil and grease/total petroleum hydrocarbons (O&G/TPH)
8. PCBs/pesticides
9. Total metals
10. Dissolved metals
11. Phenols
12. Cyanide
13. Sulfate and chloride
14. Turbidity
15. Nitrate and ammonia
16. Preserved inorganics
17. Radionuclides
18. Non-preserved inorganics
19. Bacteria.

This collection order takes into consideration the volatilization sensitivity of groundwater samples. Additional information on the order of sample collection can be found in RCRA and the *Groundwater Monitoring Technical Enforcement Guidance Document*, September 1986.

8.6.5. Overview of Passive Groundwater Sampling in Monitor Wells

Passive groundwater sampling, sometimes called "no-purge" sampling, employs devices and techniques to obtain quantitative information about the concentrations of analytes of interest from a groundwater monitoring well under ambient flow conditions. The original suggested groundwater application was for long-term monitoring at well-characterized sites (Vroblesky, 2001); however, the depth-specific nature and other characteristics of passive samples also can make them very useful for site characterization.

Passive samplers rely on the natural movement of groundwater through the screened interval of a monitor well to sample groundwater from the adjacent aquifer. In a properly constructed and developed monitor well, water in the screened interval is constantly replaced by water from the adjacent aquifer. The screened or open interval of a well does not contain stagnant water, but rather is in constant hydraulic communication with the aquifer. The rate of exchange is driven by the natural gradient of the groundwater system, and depends on the magnitude of the gradient and hydraulic conductivity of the aquifer. The well screen and sand pack of a properly designed well have a combined hydraulic conductivity greater than that of the aquifer materials, and thus provide a preferential path that concentrates flow from the surrounding aquifer through the well bore across the screened interval. Groundwater flux through the screened interval typically is greater than flow through the immediately adjacent aquifer.

Passive samplers typically are deployed on a weighted line at a specifically chosen depth within the saturated portion of the screened or open interval of a well. Care must be taken to ensure that the sampler is placed at the selected depth within the screened interval, and that the entire sampling device remains submerged for the entire deployment period.

In contrast to samples collected using a pump or conventional rigid bailer, passive sampling has negligible effect on the ambient flow in a well, and does not alter the natural steady state conditions of the water column being sampled. Induced flow, even at low-flow rates (i.e., 200 – 500 mL/minute) may change conditions in the water column, and artificially elevate turbidity by mobilizing particles that are immobile under laminar flow in the aquifer.

8.6.5.1. Comparison of Passive Samples to Pumped Samples

Passive and pumped sample collection methods each have specific characteristics that should be considered when selecting a sampling method, and when analyzing groundwater analytical data. Passive samplers collect depth-specific samples representing a very limited depth interval (typically not more than 1 to 2 feet) of the water column in the well. They represent only the water to which the sampler is exposed under ambient flow conditions. Because passive sampling does not affect flow through the well screen by pumping, purging, or otherwise mixing the water column, passive samples represent water from a confidently known, specific depth interval in the water column.

Under ambient flow, water in the sampled interval typically is dominated by horizontal movement of water across the well screen, yielding a sample that is representative of water at that depth interval in the adjacent aquifer. However, various degrees of well-specific vertical mixing are present in the screened intervals of many wells under ambient flow. In some wells, this in-well mixing obscures the details of analyte stratification in the aquifer, though in other wells, contaminant stratification in the aquifer is clearly present in the water column of the screened interval. Passive samplers have revealed vertical concentration gradients in the well water column over intervals as short as three feet. If the screened interval intersects zones of different pressure head, vertical flow through the well will result. Water in the sampled interval will be biased toward that from the zone of higher head. In wells having a screened interval greater than 5 to 10 feet, obtaining a synoptic vertical profile of concentration by spacing multiple passive samplers in vertical series should be considered for the initial passive sampling event (Vroblesky, 2001a; ITRC, 2004). If stratified, the interval of highest concentration can be monitored subsequently using a single sampler.

Pumped samples, both low-flow and high-volume purge, collect a flow-weighted sample biased toward any zones of higher conductivity intersected by, or adjacent above or below the screened interval. Also, flow induced by pumping, even at low-flow rates, can mobilize and suspend solids that are immobile under ambient laminar flow conditions in the aquifer, thereby artificially elevating turbidity. This can result in measured concentrations being affected by the sampling process, particularly for metals. Vertical flow through the well caused by the screen intersecting zones of different head also can bias the representativeness of low-flow samples, as well as that of passive samples, particularly if net vertical flow through the well bore exceeds the purge rate (sampled water will be biased toward the source of flow). Side-by-side comparisons and historical data show that concentration results obtained by low-flow sampling most often are very similar to concentration results obtained by passive sampling (ITRC, 2007). Understanding differences that may be observed often can provide valuable insight into contaminant distribution and hydrologic characteristics of a site.

8.6.5.2. Characteristics of Passive Samples

Passive samplers:

- Represent a short, discrete interval of the water column in the screened interval of a well
- Can provide a vertical concentration gradient profile of the water column and identify whether contaminant stratification is present in the well
- Improve consistency of monitoring data over time by reducing the number of variables in sample collection such as pump intake depth, adequate duration of low-flow purging, pumping rate, etc.
- Eliminate or minimize turbidity
- Greatly reduce field equipment needs thereby facilitating sampling where access is difficult, or where discretion is desired
- Eliminate purge-water production, and costs for its handling and disposal
- Can be deployed in most monitor wells (i.e., 1-inch diameter or greater)
- Have no practical depth limit

- Allow rapid field sample collection; no time is needed for purging or set up of equipment
- With few exceptions, can remain deployed in a well for up to a year without loss of integrity, allowing the retrieval of a sample and replacement of the sampler for the next round of sampling during a single site visit.

Some of these characteristics can be either an advantage or a disadvantage, depending on sampling objectives. Many of these characteristics serve to substantially reduce field labor and equipment cost, particularly for long-term monitoring programs. Field sampling cost reduction in long-term monitoring programs is typically between 40 and 70 percent (ITRC, 2004; ITRC 2007).

8.6.5.3. Limitations of Passive Sampling

Passive samplers:

- Require time for equilibration (typically up to two weeks)
- Are inappropriate for sampling where the objective is to determine water quality from a well under pumping conditions (e.g., a drinking water production well)
- Provide limited sample volume, although minimum sample volumes actually required for a single analysis by many common analytical methods are often substantially less than those routinely requested by the laboratory (see ITRC, 2007, Appendix A).

8.6.5.4. Types of Passive Samplers

Passive groundwater samplers can be grouped logically into three types, based on the operating mechanism.

Diffusion-based Samplers. Rely on diffusion of analytes from the well water into a sampler made of a semi-permeable membrane material, initially filled with deionized, analyte-free water. When deployed to a specific depth in a well, analyte concentrations in the sampler reach and maintain equilibrium with concentrations in the water to which they are exposed. The time required for equilibration depends primarily on properties of the membrane material, the diffusive characteristics of each analyte, and the temperature of the groundwater. The appropriate period of deployment also considers the time required for well flow dynamics and ambient conditions to restabilize from placement of the sampler or other disturbance. The rule of thumb is that a 14-day deployment period is ample for most types of diffusion samplers, analytes, and hydrologic conditions (ITRC, 2004; ITRC, 2007). Because analytes always diffuse across the membrane from higher to lower concentration, the samplers maintain equilibrium concentrations and track changes in concentration in the well water (within the equilibration time lag period) regardless of how long the sampler was deployed in the well. The very small pore sizes of diffusion sampler membrane materials eliminate turbidity, thus diffusion samplers represent only the truly aqueous (dissolved) concentrations of analytes.

Polyethylene Diffusion Bag (PDB) Sampler. (Vroblesky and Hyde, 1997; Vroblesky, 2001; ITRC, 2004) was the first diffusion sampler to become widely known for sampling groundwater. PDBs are constructed of low-density polyethylene tubing of 4 mil thickness, having a pore size of about 10 angstroms. They are appropriate for sampling only hydrophobic (low water solubility) VOCs, chlorinated solvents, and short chain aliphatic hydrocarbons (up to about C10). The hydrophobic nature of the polyethylene membrane makes PDBs inappropriate to sample for analytes that are highly soluble in water (e.g., ketones, alcohols, etc.) or dissolved ionic species (e.g., metal cations, anions). Tables listing many field- and laboratory-tested analytes are found in Vroblesky (2001a), Vroblesky and Campbell (2001), ITRC (2004), and ITRC (2006).

Regenerated Cellulose Dialysis Membrane Sampler. (Imbrigiotta and others, 2007) is similar in concept to the PDB, but uses a hydrophilic cellulose membrane. The dialysis membrane sampler was developed and demonstrated by researchers at the US Geological Survey (Ehlke and others, 2004) to provide a passive diffusion-type sampler appropriate for inorganic as well as organic analytes. It has been demonstrated as suitable for sampling dissolved concentrations of major cations and anions, most trace metals, all analytes on the EPA Method 8260B list, semivolatile organic compounds, many explosives, total dissolved solids, electrical conductance, dissolved organic carbon, and dissolved gases. Field and laboratory test results from dialysis membrane samplers for many analytes are listed in ITRC (2006; Tables 4-1 and 4-2). The pore size of about 18 Angstroms excludes all suspended colloidal size particles, and organic molecules >~8000 Daltons. Field-ready dialysis samplers are not currently commercially available, but can be constructed easily and economically from the component materials. One potential disadvantage of the dialysis sampler is that the cellulose membrane is subject to biodegradation, typically losing integrity in about 3 to 4 weeks in most wells, and thus cannot be deployed for extended periods (e.g., between quarterly long-term monitoring events).

Water from all equilibrium-type diffusion samplers should be transferred immediately upon retrieval to appropriate laboratory containers for transport and storage to minimize analyte loss, particularly if volatile compounds are of interest. Because diffusion through the membrane is reversible, VOC losses or other changes begin occurring as soon as the sampler is removed from the intended sampling location.

Whole-water grab samplers obtain an instantaneous sample of the water from a specific depth in the water column of a well (ITRC, 2007).

Whole-water Grab Sampling Devices. Are among the oldest groundwater sampling devices (e.g., rigid bailer). However, recent improvements in design and sampling methods provide samples from a specifically designated and confidently known depth in the water column, and recommend an equilibration period of several days to two weeks (ITRC, 2007) for both the well and sampler to reach equilibrium with ambient conditions before sample collection. Equilibration avoids analyte loss from the sample to sampling equipment, which can be significant, and allows elevated turbidity and effects of sampler placement or other disturbance to dissipate before sample collection. The sample is collected immediately prior to, or as a result of sampler retrieval. Properly collected whole-water grab samples capture suspended particles that are naturally mobile in the aquifer under ambient laminar flow conditions, but do not have elevated turbidity resulting from the sampling activity. One of the more recently developed devices seals the sample in situ at the sampling point in containers (e.g., 40-mL VOA vials or other containers) that can be placed directly into auto-samplers at the laboratory, thus eliminating all exposure of the sample to air or other influences. Sample transfer can cause loss of VOC analytes, and exposure to air can cause changes in oxidation-reduction potential affecting redox-sensitive metal concentrations.

Accumulation–type Samplers. Rely on both diffusion and adsorption of analytes. Analytes diffuse through a semi-permeable membrane and are accumulated onto an adsorbent medium within the sampler. Both the diffusion membrane and adsorbent material must be appropriate for the analyte(s) of interest. The total analyte mass accumulated on the adsorbent medium over the entire deployment period is measured. Accumulation samplers have been used to sample surface water (e.g., bioaccumulation studies) for several decades (e.g., the SPMD, Polar Organic Chemical Integrative Sampler (POCIS)) (ITRC, 2006; Verreydt, 2010), and for air and soil vapor studies (ITRC, 2006). More recently the methodology has been applied to determine VOC concentrations in groundwater wells using the Gore™ Module (ITRC, 2007). An average accumulation rate over the deployment period is easily calculated, but estimates of concentration in the medium being sampled depend on a significant number and range of analyte-specific, sampler-specific, and environmental variables (e.g., membrane and analyte diffusion properties, adsorbent uptake rate, temperature, pressure, etc.). Proprietary empirical data for the Gore Module is used to infer VOC concentrations in groundwater. Accumulation samplers have a particular advantage in that the deployment period can be extended to concentrate analyte mass and detect analytes present in the environment at concentrations too low to be detected by other sampling methods.

Tables listing media and common analytes addressed by many passive sampling devices mentioned above, advantages and limitations specific to each type of device, as well as summary of the availability and cost of each type of passive device are found in ITRC (2007, Tables 14-1, 14-2, 14-3), and in Verreydt and others (2010).

8.6.5.5. References for Passive Sampling

For more information on passive sampling, see the following references:

Ehlke, T.A., Imbrigiotta, T.E., and Dale, J.M., 2004, *Laboratory Comparison of Polyethylene and Dialysis Membrane Diffusion Samplers*, Groundwater Monitoring & Remediation, 24(1):53-59

Imbrigiotta, T.E., Trotsky, J.S, and Place, M., 2007, *Demonstration and Validation of a Regenerated-cellulose Dialysis Membrane Diffusion Sampler for Monitoring Groundwater Quality and Remediation Progress at DoD Sites*, Final Technical Report ESTCP Project ER-0313.

ITRC (Interstate Technology & Regulatory Council), 2004, *Technical and Regulatory Guidance for Using Polyethylene Diffusion Bag Samplers to Monitor Volatile Organic Compounds in Groundwater*, ITRC Technical and Regulatory Guidance, Report DSP-3, February, 2004.

ITRC (Interstate Technology & Regulatory Council), 2006, *Technology Overview of Passive Sampler Technologies*, Diffusion/Passive Sampler Team, Report DSP-4.

ITRC (Interstate Technology & Regulatory Council), 2007, *Protocol for Use of Five Passive Samplers to Sample for a Variety of Contaminants in Groundwater*, ITRC Technical and Regulatory Guidance, Report DSP-5.

Verreydt, G., Bronders, J., Van Keer, I., Diels, L., and Vanderauwera, P., 2010, *Passive Samplers for Monitoring VOCs in Groundwater and the Prospects Related to Mass Flux Measurements*, Goundwater Monitoring & Remediation, 30(2):114-126.

Vroblesky, D.A. (2001a). *User's Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells, Part 1: Deployment, Recovery, Data Interpretation, and Quality Control and Assurance*, U.S. Geological Survey Water Resources Investigations Report 01-4060.

Vroblesky, D.A., 2001b, *User's Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells, Part 2: Field Tests*, U.S. Geological Survey Water Resources Investigations Report 01-4061.

Vroblesky, D.A., and Campbell, T.R., 2001, *Equilibration Times, Stability, and Compound Selectivity of Diffusion Samplers for Collection of Groundwater VOC Concentrations*, Advances in Environmental Research, 5:1-12.

Vroblesky, D.A. and Hyde, W.T., 1997, *Diffusion Samplers as an Inexpensive Approach to Monitoring VOCs in Groundwater,* Groundwater Monitoring and Remediation, 17:177-184.

8.7. In Situ Groundwater Sampling Using Probes

This section provides a description of the equipment and techniques that can be used to collect samples using probes rather than monitoring wells. It is important to check with state environmental agencies to determine if groundwater data collected from probes can be used for decision-making purposes or site closure.

A relatively new development in groundwater sampling technology has been the design of in situ sampling probes, which allow for the collection of groundwater samples without the installation of permanent wells. A hydropunch operates in conjunction with conventional cone penetrometer rigs. The category of in situ sampling probes also includes a variety of driven probes, which can be retrieved after sampling or left in place as permanent sampling points. These devices often are used during the preliminary site characterization stage or where only a shallow water table is to be sampled. The portable in situ samplers can be valuable in determining the best location for permanent monitoring wells.

CAUTION: Not all states accept groundwater data collected with probes. Site Managers should consult state environmental agencies when developing the SAP to determine how groundwater data from probes can be used.

8.7.1. In situ Well Installation

Direct push technology (DPT) is an example of an in situ groundwater sampling tool that can be used to install point-in-time groundwater sampling probes and wells. A description of some DPT technologies is provided below.

8.7.1.1. Point-in-Time Groundwater Sampling Probes

Point-in-time probes, also referred to as “temporary samplers” or “grab samplers,” are used to collect samples rapidly to define groundwater conditions during one sampling event. They are usually less than 2 inches in outside diameter (OD) and are generally constructed of steel or stainless steel. Direct-push methods (percussion or static weight) are used to advance point-in-time samplers below the static water level in unconsolidated formations. Generally, groundwater flows into the sampler from an exposed screen under ambient hydrostatic pressure. Groundwater may be collected from the sampler using bailers or pumps, or the sampler may be retracted to the surface to obtain the water sample. Once sampling is completed, these devices are removed and the boring should be abandoned in accordance with local regulations.

8.7.1.2. DPT-Installed Groundwater Monitoring Wells

DPT-installed monitoring wells permit short-term or long-term monitoring of groundwater. They are usually 2 inches in diameter or less and constructed of PVC or stainless steel. Because monitoring wells are installed for periods of several months to several years, the annulus of the boring around the well casing is usually sealed to prevent migration of contaminants into the aquifer. Surface protection is required to prevent tampering with the well. A slotted or screened section permits groundwater to flow into the well under ambient hydrostatic pressure. Groundwater may be collected from monitoring wells using bailers, various pumps, or passive sampling devices.

8.7.2. Point-in-Time Sampling

Ideally, DPT point-in-time and monitoring well groundwater sampling equipment should be used together to maximize their effectiveness. Point-in-time sampling techniques generally are better for identifying plume boundaries, hot spots, preferred pathways, or other monitoring points of interest.

A variety of point-in-time groundwater sampling tools are available for site characterization, including the following:

- Sealed-screen samplers
- Multi-level samplers (or vertical profilers)
- Open-hole samplers.

With these tools, the time needed to retrieve the sample will vary according to the hydraulic conductivity of the sampling zone. In general, sampling within coarse-grained sediments takes minutes, while sampling within fine-grained sediments can take several hours or more. In situations where slow recharge inhibits the timely collection of groundwater samples, the sampler may be left in place to recharge while the DPT rig is moved to a new sampling location.

8.7.2.1. Sealed-Screen Samplers

Sealed-screen samplers typically consist of a short (6 inch – 3-foot) screen nested within a sealed, watertight tool body. Because the screen is not exposed to the formation as the sampler is advanced into the subsurface, the screen does not become plugged or damaged. In addition, the potential for cross-contamination is greatly reduced and a true depth-discrete sample that is representative of the target sampling zone can be collected. The sample volume collected with some sealed-screen samplers is limited by the volume of the sample chamber.

To collect the sample, the sealed-screen sampler is advanced to the target sampling depth, and the protective outer rod is retracted, exposing the screen to groundwater. Groundwater flows through the screen and into the drive rods or sample chamber under the hydraulic head conditions that exist at that depth. O-ring seals placed between the drive tip and the tool body help ensure the sampler is watertight as it is driven to the target sampling interval. The integrity of the seal often can be checked by lowering an electronic water level indicator into the sampler prior to retracting the protective outer rod.

8.7.2.2. Multi-level Samplers

Multi-level samplers, most of which are exposed-screen samplers, are DPT equipment capable of collecting groundwater samples at multiple intervals as the sampling tool is advanced. One benefit is they do not have to be withdrawn for sample collection or decontamination. The terminal end of a typical multi-level sampling tool has a 6 inch – 3 foot-long screen made up of fine mesh, narrow slots, or small holes. The screen remains open to formation materials and water while the tool is advanced. Samples are collected either continuously or periodically as the tool is advanced to vertically profile groundwater chemistry and aqueous-phase contaminant distribution.

Multi-level samplers can be used to measure water levels at discrete intervals within moderate- to high-yield formations to assist in defining vertical head distribution and gradient. Additionally, some of these tools can be used to conduct hydraulic tests at specific intervals to characterize the hydraulic conductivity in formation materials and identify possible preferential flow pathways and barriers to flow.

8.7.2.3. Open-hole Sampling Methods

Open-hole sampling is conducted by advancing drive rods with a drive point to the desired sampling depth. Upon reaching the sampling depth, the rods are withdrawn slightly, which separates them from the drive tip and allows water to enter the rods. The water can be sampled by lowering a bailer into the rods or by pumping. The open-hole method is feasible only within formations that are fairly cohesive; otherwise, the formation may flow upwards into the rods when they are withdrawn, preventing samples from being collected. With single-rod systems, open-hole sampling can be conducted only at one depth within a borehole because the borehole cannot be flushed out between sampling intervals, which could cause cross-contamination. Dual-tube systems, on the other hand, can be used to conduct multilevel sampling.

8.7.3. DPT Monitoring Well Installation

A variety of DPT methods are available for installing temporary or permanent monitoring wells. The two main installation methods used are exposed-screen and protected-screen wells. These methods are discussed in detail in ASTM D6724 and D6725, and are summarized here. As with conventional well installations, hydraulic connections should not be created between otherwise isolated water-bearing strata. In addition, precautions should be taken to minimize turbidity during the installation of filter packs and the development and sampling of DPT wells.

8.7.3.1. Exposed-screen Well Installation Methods

With exposed-screen well installation methods, the well casing and screen are driven to the target depth using a single string of rods. Because the screen is exposed to formation materials while it is advanced, proper well development is important to remove soil from screen slots. This method is not recommended for installing well screens within or beneath contaminated zones because drag-down of contaminants with the screen may cross-contaminate sampling zones and can prevent the acquisition of samples representative of the target zone. Exposed-screen well installation methods should be used only in upgradient areas that are known to be uncontaminated. Also, some states prohibit the practice of allowing the formation to collapse around a well screen during the construction of a monitoring well. Therefore, state regulations should be consulted before selecting exposed-screen techniques.

8.7.3.2. Protected-screen Well Installation Methods

When installing a protected-screen well, the well casing and screen are either advanced within or lowered into a protective outer drive rod that has already been driven to the target depth. Once the well casing and screen are in place, the drive rod is removed. Alternatively, the casing, screen, and a retractable shield may be driven simultaneously to the target depth. Once in place, the screen is exposed and the entire unit remains in the ground. If there is sufficient clearance between the inside of the drive rod and the outside of the well casing and screen, a filter pack and annular seal may be installed by tremie (an apparatus for depositing and consolidating concrete under water) from the surface as the drive casing is removed from the hole. Several filter packing and annular sealing approaches are available, depending on the equipment used for the installation. Regardless of the method of installation, the filter pack should be sized appropriately to retain most of the formation materials.

The most common protected-screen method for installing DPT wells is to advance an outer drive casing equipped with an expendable drive tip to the target depth. The well casing and screen are then assembled, lowered inside the drive casing, and anchored to the drive tip. The drive casing seals off the formations through which it has been advanced, protecting the well casing and screen from clogging and from passing through potentially contaminated intervals. The position and length of the screen should be selected to match the thickness of the monitoring zone, which can be determined by using additional information, such as cone penetration testing (CPT) logs or continuous soil boring logs.

8.8. Sample Handling and Collection

This section provides guidance on sample handling procedures, such as filtration, and specific techniques for collection of samples that will be analyzed for various classes of groundwater pollutants, such as SVOCs. For detailed sampling procedures and preservation of samples, refer to Appendix B of this handbook. It is important to remember that COC procedures should be followed for all groundwater sampling events and that all samples (except for metals) remain on ice during both sample collection and shipment to the laboratory. Details regarding the use of COC procedures are provided in Chapter 3.

8.8.1. Sampling for Metals

Certain regulations require metals analyses to be performed on unfiltered groundwater samples. The reason for these requirements is to obtain a representative sample as it actually occurs in the aquifer and to maintain consistency between sample handling for inorganic and organic analyses. If a particular situation demands consideration of dissolved metals, both filtered and non-filtered samples should be collected for analysis. The regulatory document or approved SAP should be consulted for monitoring requirements.

The differences obtained as a result of sample handling (filtered versus non-filtered) are dependent on the type of association between the specific inorganic ion and the particulate matter. Studies show that when an inorganic ion is not closely associated with particulate matter (e.g., sodium), the differences between total and dissolved concentrations are small and random.

Ideally, the sample can be split into two portions, one for filtration and the other for immediate preservation and subsequent analysis for total metals concentration. By analyzing the two fractions separately, differences between dissolved and total metals can be compared.

The decision of whether to filter metals samples should be based on the physical quality of the samples, the objectives of the monitoring program, and the policy of the regulation or agency controlling the sampling event. If filtering is allowed and chosen, it is imperative that it be performed in a manner that will preserve the integrity of the sample and allow for consistent reproduction of technique.

Filtration of groundwater samples for dissolved metals analyses should be performed with a pre-cleaned filtering apparatus. Sampling devices should be cleaned using ultra-pure nitric acid when low-level metals contaminants are being measured. Filtration must be done immediately upon sample collection, prior to preservation. Samples transported to the lab for filtration and preservation should be documented, because sample composition will change during transport. The sample should be collected and filtered through a 0.45-µm pore diameter cellulose acetate filter. If using a vacuum filter is impractical, pressure filtration must be performed. Care should be taken to follow the manufacturer's recommended procedure if vacuum filtration is used. All filter apparatus should be laboratory-cleaned and dedicated; however, disposable filters are acceptable. For each sampling event, a new disposable filter must be used to avoid cross-contamination of samples.

Groundwater samples to be analyzed for both total metals and dissolved metals are collected in plastic bottles and preserved with nitric acid to pH < 2. All appropriate data should be recorded in the FLB/FN.

8.8.2. Sampling for VOCs

At each site, samples for analysis of VOCs are collected first. Care should be taken to prevent volatilization of the sample when placing groundwater into the vial. Groundwater samples collected for analysis of VOCs, either from passive diffusion bags, low-flow pumps, or bailers, must be placed into 40-milliliter vials with a septum.

A sacrificial 40-milliliter sample should be obtained to determine preservation requirements before VOC samples are collected. Fill the vial with sample water, then add 1:1 HCl, drop-by-drop, and test the pH until it is < 2. If the test sample effervesces when the HCl is added, the samples should not be acid preserved and should be analyzed within 7 days of collection. Record the amount of HCl added. Alternatively, prepreserved VOC vials may be obtained from the analytical laboratory.

The procedure for collecting a VOC sample is described below:

1. Remove the cap from a 40-mililiter septum (Teflon®-faced silicon rubber) vial. Avoid contact with the inner surface.
2. Add the established amount of HCl necessary for sample preservation (if prepreserved vials have not been supplied by the laboratory).
3. Fill the vial with sample water, taking care to minimize aeration, and screw on the cap tightly to achieve zero headspace. The Teflon®-lined side of the cap must face the aqueous sample.
4. Inspect the vial for air bubbles. If air bubbles are present, discard the vial and restart with step 1.
5. Label the vial, and place it in a resealable bag.
6. Record appropriate data in the FLB/FN.

8.8.3. Sampling for Extractable Organics

The procedure for collecting groundwater samples for analysis of SVOCs is described below.

1. Remove the Teflon®-lined cap from a 1-liter amber glass bottle. Avoid contact with the inner surface of the cap.
2. Fill the bottle with groundwater.
3. Replace the cap tightly, attach the sample label, and place the sample bottle in a cooler with enough bagged ice to cool the sample to ≤ 6°C.
4. Repeat steps 1 through 3 for additional samples.
5. Record appropriate data in the FLB/FN.

8.8.4. Sampling for Cyanide

The procedure for collecting groundwater samples for analysis of total cyanide is described below.

1. Place groundwater into a 1-liter wide-mouth plastic bottle.
2. Determine if sulfide is present. Pour a small aliquot of sample from the 1-liter wide-mouth plastic bottle into a clean 100-mililiter beaker and test for sulfide using a lead acetate test strip. If sulfide is:
   - Present and visible solids are present, go to step 3
   - Present and no visible solids are present, go to step 5

- Not detected, go to step 8.

3. If sulfide is present and visible solids are present, filter the sample through a 0.45-µm cartridge filter within 15 minutes of collection. The sample should be pumped through the filter using new Tygon® tubing into a second clean, 1-liter wide-mouth plastic bottle.
4. Place the spent 0.45-µm cartridge filter in a 1-gallon resealable bag, label the bag with the appropriate sample number and collection time, and place the filter cartridge and bag into the freezer. The frozen filter can be sent to the laboratory for analysis of cyanide.
5. For the presence of sulfide, add 1 gram of solid lead carbonate to the 1-liter wide-mouth sample bottle, cap the bottle, and mix by inverting the bottle several times.
6. Check for the removal of sulfide. Pour a small aliquot of lead carbonate-treated sample into a clean 100-milliliter beaker and test for sulfide again using the lead acetate test strip. If sulfide is present, add another gram of lead carbonate to the sample, cap the bottle, and mix by inverting the bottle several times. Repeat this step until sulfide is not detected by the lead acetate test strip.
7. Filter the lead carbonate-preserved sample through a new 0.45-µm cartridge filter to remove residual chemical addition and lead sulfide formed during the precipitation reaction. Collect the filtrate in a clean 1-liter plastic wide mouth bottle. Discard the 0.45-µm cartridge filter.
8. Determine if total chlorine is present. Pour a small aliquot of sample from the 1-liter wide-mouth plastic bottle (preserved, if sulfide was present) into the appropriate vials of the chlorine test kit and analyze for total chlorine. If total chlorine is present in the sample, add 0.1 gram of sodium borohydride ($NaBH_4$), cap the bottle, and mix by inverting the bottle several times. If chlorine is not detected, go to step 10.
9. Check for the removal of chlorine. Pour a second small aliquot of $NaBH_4$-treated sample into the appropriate vials of the chlorine test kit and analyze for total chlorine. If chlorine is present, add another 0.1 gram of $NaBH_4$. Repeat this step until chlorine is no longer detected. Generally, 0.1 gram/liter $NaBH_4$ will reduce 50 mg/liter of chlorine.
10. Adjust the pH of the sample to > 12 using sodium hydroxide (NaOH), and place the sample on ice to lower the temperature to ≤ 6°C prior to shipping to the laboratory. When the sample has cooled, pack and ship using normal procedures. Record all preservation procedures in the FLB/FN.

8.8.5. Sampling for Conventional Parameters and Nutrients

Conventional pollutants typically include BOD, O&G/TPH, TOC, COD, TDS, TSS, and chloride. Nutrients include ammonia, nitrate, and total phosphorous. Bottle and preservation requirements will differ based on each particular analyte. The procedure for collecting groundwater samples for analysis of conventional pollutants and nutrients is described below:

1. Remove caps from sample bottles. Avoid contact with the inner surface.
2. Fill bottles to about 90 percent full. Add appropriate preservative to the samples per Appendix B of this handbook or the SAP.
3. Replace and tighten caps, attach labels, seal in resealable bags, and place sample bottles in a cooler with enough bagged ice to cool the samples to ≤ 6°C.
4. Record all appropriate data in the FLB/FN.

8.8.6. Sampling for Radioactive Elements

Groundwater samples typically are analyzed for the following radioactive constituents: gross alpha, gross beta, tritium, strontium-90, combined radium-226/228, and uranium. Other radioactive elements, such as plutonium, cesium-137, or americium-241, also may be included in groundwater sampling programs, depending on the historical uses at the site. The types of sample bottles and the required volume of the groundwater sample will vary depending on the radioactive element. The SAP must contain information on sample bottles, sample preservation, and sample holding times for specific radioactive elements that will be analyzed. When preparing the SAP, refer to the Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) guidance on field sampling of radioactive analytes (http://www.epa.gov/radiation/docs/marlap/402-b-04-001b-10-final.pdf).

Note: Shipment of radioactive samples to the laboratory may require special handling procedures. Samples to be shipped off site must be field-screened to determine the levels of radiation present. The screening will allow for proper packaging and shipping via International Air Transport Association (IATA) Dangerous Goods Regulations. By IATA definition, a material with an activity level of more than 2,000 pCi/g is considered radioactive. If groundwater samples exceed this level, special IATA packaging procedures are required for shipment by air. Packaging procedures for radioactive samples must be included in the SAP, and special training of field personnel may be required for the handling and packaging of radioactive groundwater samples.

8.8.7. Sampling for Light, Non-aqueous Phase Liquids

Light, non-aqueous phase liquids (LNAPLs) are generally considered to be low density, immiscible organics, including gasoline, petrochemicals, and other chemicals with specific gravities of less than water. They are usually present in aquifers as a separate phase due to their low solubility in water. These chemicals tend to float on the water surface in a water table environment and commonly occupy the capillary fringe zone above the water table. If LNAPLs are suspected to be floating on the water table, all shallow wells installed in the area under investigation must be screened across the water table.

Note: Samples of the floating organic layer must be collected before the well is purged.

In a confined aquifer, LNAPLs are found along the upper surface of the permeable material and also within the overlying confining layer. When immiscible organics with a specific gravity less than water are the contaminants of concern, or if contaminants are suspected in more than one stratified layer in the well column, sampling procedures must be modified. It may be necessary to lower the sampling bailer to a particular depth in the well or to utilize a double check-valve bailer. Sampling procedures for LNAPLs differ substantially from those for other pollutants. If more than one distinct LNAPL layer is present in a well, each layer should be sampled. Samples should be analyzed for chemical composition (e.g., for VOCs and base-neutral extractables) and physical parameters (e.g., specific gravity, water solubility, vapor pressure of the liquid, and Henry's Law Constant).

Measurement of the thickness of the floating layer can be accomplished by using a water indicator paste or gel with a weighted steel tape to determine the depth to the top of the floating layer and to the water surface. The difference between these two readings is the thickness of the floating layer. Measurement of the thickness of the floating layer also may be accomplished by using an interface probe or a clear Teflon® bailer (if the product thickness is less than the length of the bailer). Electric water level sounders will not work properly for these determinations.

After a monitoring well is initially constructed, it should be developed and pumped to remove stagnant water. Then the well should sit idle for at least 2 weeks to allow the water level and floating layer(s) to stabilize fully. Prior to purging the monitoring well, a sample of the floating layer can be obtained using a bailer that fills from the bottom.

The collection of a water sample from a well containing free-floating product is challenging and at times problematic. This is true even if the LNAPL floating in the well has been removed during the purging process (the LNAPL can still coat the inside casing, or be entrained in the screen and sand pack, and likely is emulsified below the air/water interface). Care should be taken to lower the bailer just through the floating layer but not significantly down into the underlying groundwater. Care also should be taken in proper planning when sampling water from such wells and interpreting the resulting data (e.g., to determine if the sample is representative of the actual groundwater concentrations). Various sampling methods may be used to provide greater confidence in the results of the analysis. Stilling tubes may be used to help prevent the sampling device from contacting residual LNAPL that can impact the collected water sample. Discrete interval samplers also may be used to ensure that emulsified product does not enter the sampler during emplacement below the water level in the well. Samples should be analyzed to determine the chemical composition and physical properties of the LNAPL. After following typical evacuation procedures discussed previously in this section, a sample of formation water may be obtained from the well.

8.8.8. Sampling for Dense, Non-aqueous Phase Liquids (DNAPLs)

DNAPLs include chlorinated solvents and other chemicals with specific gravities greater than water. They are usually present in aquifers as a separate phase due to their low solubility in water. DNAPLs tend to migrate downward through both the unsaturated zone and the saturated zone due to their high density. If the volume of the DNAPL introduced into the subsurface is larger than the retention capacity of the vadose and saturated zones, a portion of the DNAPL will spread out as a layer of free liquid on the bottom of the aquifer or on lower permeability beds within the aquifer.

Measurement of the thickness of DNAPLs must be performed prior to purging the well. Measurement of the DNAPL can be accomplished by using a water indicator paste or gel with a weighted steel tape (if no LNAPL is present) to determine the depth of the top of the DNAPL and the bottom of the well. The difference between these two measurements is the thickness of the DNAPL. An interface probe also may be used to measure DNAPL thickness in the well.

Note: Samples of the settled organic layer must be collected before the well is purged.

Prior to purging the monitoring well, a sample of the DNAPL can be obtained using a dual check-valve bailer or a bladder pump. If both LNAPLs and DNAPLs are present, it may be necessary to purge the well of one casing volume of water prior to sampling the DNAPL. Efforts should be made not to disturb the DNAPL in the bottom of the well by setting the pump intake of the submersible or suction-lift pump several feet above the DNAPL.

Samples should be analyzed to determine the chemical composition of the DNAPL and its physical properties (e.g., specific gravity, water solubility, equilibrium vapor pressure of the liquid, and Henry’s Law Constant). After the well is purged, a sample of the groundwater may be obtained for laboratory analysis.

8.9. Sampling Industrial Wells

This section provides a brief discussion on sampling industrial wells used to extract process water and potable water. Site Managers should always consult the state environmental agencies drinking water division to determine specific requirements.

When sampling industrial wells, it is desirable to sample as close to the well source as possible. Samples should be taken directly from the well head whenever possible to eliminate treatment interferences, possible changes in quality within the lines, and mixing of water from other wells.

Large-capacity wells that are “on-line” during monitoring can be sampled immediately. Wells that are “off-line” must be purged and pumped to waste prior to sampling. Fifteen minutes or more of pumping is suggested. Access to municipal well systems, well houses, and other related structures will require the assistance of the site manager or water department employee. Prior notification is essential.

8.10. Aquifer Testing

At sites with contaminated groundwater, aquifer testing may be performed to determine site-specific aquifer properties that help define the transport of groundwater and contaminants. If groundwater remediation is appropriate, an aquifer pumping test may be performed. During a pumping test, groundwater is removed from the aquifer for a period of several hours or days. Sampling of the extracted water is required to determine its disposal requirements. Samples are typically collected from the containment vessel using a bailer, or directly filling sample bottles from a port on the container.

Long-term aquifer tests can be used to estimate contaminant loading on treatment systems by collecting samples from a sample tap in the pump discharge pipe at various times over the duration of the aquifer test. This provides a more realistic estimate of treatment system loading than using individual samples collected from monitoring wells. Use of monitoring well samples alone can result in an overestimation of contaminant loading (and overdesign of the treatment process) if groundwater concentrations decrease over the course of the pumping test.

8.11. Well Decommissioning or Abandonment

This section provides an overview of the requirements for well decommissioning and abandonment. It is important that Site Managers check with state environmental agencies to determine specific requirements.

Unsealed or improperly sealed groundwater monitoring wells, including DPT wells, may threaten public health and safety and the quality of groundwater resources. Proper well abandonment accomplishes the following:

- Eliminates the physical hazard of the well (the actual hole in the ground)
- Eliminates a pathway for migration of contamination
- Prevents hydrologic changes in the aquifer system, such as the changes in hydraulic head and the mixing of water between aquifers.

The proper decommissioning method will depend on both the reason for abandonment and the condition and construction details of the boring or well.

Typically, well abandonment is the responsibility of the property owner and must be done according to state regulations. In the absence of more stringent regulatory standards, the procedures outlined below represent minimum guidelines for proper abandonment of wells and borings. These steps may be applicable, but not limited to, public and domestic water supply wells, monitoring wells, borings, or drive points drilled to collect subsurface information, test borings for groundwater exploration, and dry wells (drains or borings to the subsurface).

1. The well should be plugged to prevent the entrance of surface water, circulation of water between or among producing zones, or any other process resulting in the contamination or pollution of groundwater resources.
2. The well should be chlorinated prior to abandonment using a chlorine solution with a minimum concentration of 150 ppm.
3. The entire depth of the well should be checked before it is sealed to identify any obstruction that could interfere with sealing operations.
4. The well bore should be filled and sealed completely with bentonite cement grout.
5. The grout material should be placed in a way that prevents voids in the grout or dilution of the grout.
6. The abandoned well or bore should not become a channel for the vertical movement of water or other substance to potable groundwater resources.
7. Upon completion of well abandonment, the top of the casing and grout material should not be terminated more than 4 feet below the ground surface of the final grade.

8.12. Sampling Equipment List

Section 3.8 of this handbook provides a generic sampling equipment list applicable to most sampling events. The following list provides additional equipment applicable to groundwater sampling. In most cases, only a portion of the equipment listed below would be required for a given sampling event.

- Water level indicator
- Steel line and chalk
- Electric tape (e.g., interface probe, slope indicator, M-scope)
- Well evacuation equipment
  - Suction lift pump/centrifugal pump
  - Portable submersible pump

  - Peristaltic pump
  - Air lift pump
  - Bladder pump (gas squeeze pump)
  - Packer pump
  - Gas piston pump
  - Gas displacement pump
  - Inertial pump
- Groundwater sampling equipment
  - Passive diffusion bags for VOCs
  - Bottom fill bailer (single or double check valve)
  - Peristaltic pump
  - Bladder pump
  - Packer pump
  - Inertial pump
  - Syringe sampler
- Additional equipment
  - VOCs detection devices, such as PID or FID
  - Appropriate hand tools
  - Keys to locked wells
  - Filtering devices
  - Field measurement instrumentation (e.g., temperature, specific conductance, pH, DO, turbidity)
  - Plastic sheeting
  - Dedicated, pre-cleaned stainless steel pitchers, or an equivalent dipping device
  - Calibrated bucket for purged water measurement
  - Distilled or deionized water or ASTM Type II water
  - Laboratory-grade glassware, detergent, or cleaning materials
  - Empty drums for collection of purged water
  - Stainless steel clamps.

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Chapter 9. Drinking Water Sampling

9.1. Purpose

This chapter provides standard drinking water sampling procedures that accurately represent the condition of drinking water and comply with federal guidelines.

9.2. Background

The SDWA was passed in 1974 to protect public health by regulating the nation's public drinking water supply. The law was amended in 1986 and 1996 to expand its scope and increase the number of regulated contaminants. The SDWA authorizes EPA to set national health-based standards to minimize the concentration of harmful contaminants in drinking water and protect the population from naturally occurring or manmade pollutants. Currently, there are 96 drinking water contaminants regulated under the SDWA.

The SDWA applies to every public water system (PWS) in the United States. There are more than 170,000 PWSs that provide piped water for human consumption in the United States. A PWS is defined as having at least 15 connections or regularly serving at least 25 customers for at least 60 days per year. PWSs are classified as either community water systems (CWSs), which number about 60,000, or non-community water systems (NCWSs), which number about 110,000. A CWS supplies water to the same population year round while a NCWS is defined by the population served (e.g., transient or non-transient). A transient non-community water system (TNCWS) serves the public, but not the same individuals, for more than 6 months, such as in campgrounds. A non-transient non-community water system (NTNCWS) serves the public and the same population for more than 6 months, but not year-round, such as in schools. The majority of the population in the United States receives drinking water from a CWS. Household wells are exempt from regulation, but the recent promulgation of the Ground Water Rule (GWR) incorporates all PWSs that use groundwater as a source.

While a TNCWS is monitored for a few contaminants (e.g., bacteria, nitrate, and nitrite), CWS and NTNCWS monitoring is more extensive. The breadth of monitoring required by the SDWA is dependent on the section of the drinking water system identified (e.g., source water, treatment, distribution system, or tap), the number of customers, and the source of the water. Source water (i.e., groundwater, surface water, or groundwater under the influence of surface water) and the point-of-entry into a water treatment system have modest monitoring require-ments that are usually handled by annual sampling for inorganic chemicals, nitrate, nitrite, radionuclides, synthetic organic chemicals (SOCs), VOCs, and asbestos. As the drinking water progresses through the distribution system, the microbiological monitoring requirements increase, as does the analysis for residual disinfectant and contaminants related to treatment and conveyance (e.g., trihalomethanes (THMs), lead, and copper). For a utility serving more than 10,000 customers, as many as 50 microbiological samples per month can be required. Section 9.7.2.4 indicates the number of samples that would be required for smaller PWSs. To learn the exact monitoring requirements for a particular drinking water system, refer to 40 CFR 141 or the EPA *Interactive Sampling Guide for Drinking Water System Operators* (http://www.epa.gov/safewater/smallsys/samplingcd.html), or consult state drinking water program coordinators.

The National Primary Drinking Water Regulations (NPDWRs) specify health-based, enforceable maximum contaminant levels (MCLs) for specific pollutants found in drinking water or require treatments to remove contaminants to a safe level. The MCL is the concentration of pollutant that will have no known or expected risk to human health if consumed in drinking water. Utilities are required to test for each applicable pollutant and ensure that the MCLs are being achieved. The SDWA, as amended and codified in CFR 141.151, requires utilities to provide an annual report to customers attesting to the condition of the water with the associated MCLs.

National Secondary Drinking Water Regulations (NSDWRs) specify non-enforceable levels of contaminants in drinking water that are intended to mitigate suspected but unproven health risk consequences, and conservatively serve as a goal that providers of drinking water should strive to meet maximum contaminant level goals (MCLGs). MCLGs can carry some public health considerations but also carry aesthetic characteristics such as taste and odor. An action level (AL) is the maximum concentration of a contaminant that would have no adverse health effect if consumed in drinking water. An AL is health-based and, like an MCL, prompts aggressive action by the utility to reduce the concentration of the pollutant to safe levels. Usually ALs are related to the treatment techniques applied to drinking water.

Because of the SDWA, EPA has introduced a number of rules to address specific problems related to drinking water safety. The titles of most of the rules are self-explanatory and result in some additional monitoring and reporting. The drinking water safety rules developed by EPA are as follows.

- **GWR.** Focuses on increased protection from microbes in ground water.
- **Total Coliform Rule (TCR).** Tightens the SDWA's requirements on the concentration of bacteria in treated water because positive coliform tests can indicate treatment problems.
- **Surface Water Treatment Rule (SWTR).** Addresses the control of *Giardia* and viruses. Subsequent revisions include *Cryptosporidium* disinfection or removal.
- **Lead and Copper Rule (LCR).** Deals with potentially toxic levels of metals derived from plumbing materials in the distribution system and ways to minimize their concentration. Tests that exceed the AL for lead or copper can trigger extensive home monitoring.
- **Arsenic Rule (AR).** Reduces the MCL for arsenic from 50 µg/L to 10 µg/L. Additional treatment could be needed to meet the new MCL.
- **Disinfectants and Disinfectant Byproducts Rule (DDBPR), Stage 1 and Stage 2.** Regulates the halogenated chemicals produced by the disinfection of drinking water. THMs, haloacetic acids (HAA5), Bromate, and Chlorite are common disinfection byproducts (DBPs) regulated by the EPA.
- **Unregulated Contaminants Monitoring Rule (UCMR).** Preserves the public health of citizens by identifying new or emerging pollutants associated with source water, drinking water treatment, distribution systems, and consumption.
- **Interim Enhanced SWTR.** Created in February 1999 to improve the control of microbial populations, particularly *Cryptosporidium*, while balancing the risks from the production of disinfection by-products.
- **Long-Term Stage 1 SWTR.** Became effective in January 2002 and built on the control of microbial populations, specifically *Cryptosporidium*. It strengthened the filtration requirements, implemented the Stage 1 DBP Rule, and incorporated smaller water systems.
- **Long-Term Stage 2 Enhanced SWTR.** Became effective in 2006 as a further extension of the *Cryptosporidium* and microbial control requirements of the earlier rules. All systems using surface water or water directly under the influence of surface water are included. The rule requires routine sampling for *Cryptosporidium*. Treatment depends on the levels of *Cryptosporidium* reported.

All of the sampling and analytical requirements associated with the recently promulgated rules are included in the EPA Interactive Sampling Guide for Drinking Water System Operators and on EPA's website, http://www.epa.gov/safewater.

9.3. Scope

This chapter provides the following procedures for sampling groundwater and surface water supplies for monitoring drinking water quality:

- Microbiological, inorganic, and organic sampling

- Monitoring of metals at the tap
- Monitoring of turbidity levels for surface water supplies.

To meet the requirements of the SDWA and the related contaminant regulations, as well as ensure the quality of a drinking water supply, a comprehensive drinking water sampling program must be developed. The drinking water sampling program should include detailed information about the water source, entry point, treatment process, and distribution system. This information will help samplers determine which monitoring requirements apply to a particular drinking water supply. Monitoring requirements for source water sampling or point-of-entry sampling can be modest when compared with monitoring requirements for points further along in a distribution system, or for a system serving a large population. The drinking water sampling program must consider the monitoring requirements for each component of the water distribution system to ensure that all applicable regulations are met. A comprehensive drinking water sampling program should compare samples collected from the source water, entry point after treatment, and distribution system to determine the effect of treatment and plumbing systems on drinking water quality.

9.4. Project Planning

This section describes the steps in planning a drinking water sampling program. Before each drinking water sampling program can begin, concise objectives must be developed regarding the intent of the sampling program. The objectives provide the framework for developing a sampling strategy and preparing the sampling and analysis plan.

9.4.1. Sampling Strategy

The sampling strategy will help the project manager determine the appropriate sampling locations, sample numbers, and sample frequency based on the following:

- Water source
- Source vulnerability
- Analyses to be performed
- Regulatory requirements
- DQOs
- The population served
- Anticipated or potential contaminants
- System size.

9.4.2. Site-Specific SAP

A site-specific SAP should be prepared and reviewed by appropriate regulatory personnel prior to starting any sampling event to ensure the proper location, parameters, and number of samples are taken to meet compliance requirements. The specific monitoring and reporting requirements of the SDWA are beyond the scope of this handbook.

The site-specific SAP is an instruction manual for field personnel. It should be built around the objectives and sampling strategy. The SAP must consider a variety of factors including the water source, condition and accessibility of sampling locations, suspected contaminants, analytical methods and detection limits, collection methods, sample handling procedures, and safety. See Chapter 2 for a detailed discussion of SAP development applicable to all sampling programs.

Note: Site-specific sampling requirements are unique, and local, state, and federal regulatory authorities should be consulted to ensure the drinking water sampling program is adequately prepared.

The SAP cannot be implemented effectively if field personnel do not understand its contents. Field personnel should fully understand the SAP, and additional training should be provided prior to field activities, if necessary.

9.4.3. Certification

Some states might have certification programs that require drinking water samples collected for SDWA compliance be collected by approved individuals. Some states also have certification training programs and require samplers to be certified for collecting drinking water samples. Local health and environmental departments require that water samples be collected in accordance with federal, state, or local regulations. A review of the sampling plan must ensure that the samples collected will meet the necessary local and state requirements for sampling personnel.

Only laboratories that are certified by the local, state, or federal program with jurisdiction are allowed to perform compliance testing for microbiology, inorganic, organic, and radiochemistry analytes. However, any person acceptable to the state can perform analyses for turbidity, chlorine residual, and pH if primacy has been granted.

EPA- or state-certified laboratories should supply the blanks, containers, and preservatives for sampling. These must be free of the contaminants at the detection levels. These materials should be traceable and documented as free of contaminants to aid the data reviewer and to avoid resampling due to avoidable contamination.

9.4.4. Analytical Methods

When taking samples for regulatory compliance, EPA-approved methods must be used. Approved methods are listed in 40 CFR 141 and Appendix B of this handbook.

9.4.5. Quality Assurance

Use the following protocol to ensure integrity and accuracy of the data collected during drinking water sampling. The laboratory analysis must be performed by a certified laboratory for the method required.

All samples must be accompanied by a complete COC record. Field blanks, trip blanks, and field duplicates can be collected as part of the QA plan to enable data evaluation for accuracy and integrity of drinking water sampling. QA/QC varies with the regulatory requirements and site location for each sample.

Detailed information on QA/QC, including the systematic planning process, development of DQOs, and preparation of QAPPs, is available in Chapter 2 of this handbook.

9.4.5.1. Trip Blanks

Trip blanks (or transport blanks) should be prepared by the laboratory and usually consist of two laboratory-filled vials in each container used to ship water VOC samples. The trip blank is analyzed to show that contamination does not occur during transportation of the VOC samples to the laboratory.

9.4.5.2. Field Blanks

Field blanks are samples of reagent water that are transferred from one vessel to another at the sampling site. Typically, the laboratory will fill sample bottles with ASTM Type I or II water, seal the bottles, and ship them to the sampling site along with the empty bottles. The field crew will transfer the reagent water to another set of clean sample bottles in the field and transport or ship the field blanks with the actual samples to the laboratory. This blank is used to show that the sampling procedures and atmosphere at the sampling site have not caused contamination. Additional reagent water should be available in field to ensure no air bubble in field blanks.

9.4.5.3. Field Duplicates

Field duplicates are collected from field-homogenized samples, stored in two separate containers, and analyzed separately at the same laboratory. For VOCs, field duplicates are two separate samples that are collected as close as possible to the same point in space and time, stored in separate containers, and analyzed separately at the same laboratory. These duplicates are useful in documenting the precision of the sampling process.

9.5. Hazards and Safety Precautions

A site-specific HASP that addresses all anticipated hazards should be prepared for each task. Specifically, the HASP should address the following hazards that could be associated with drinking water sampling:

- Exposure to biological and chemical contaminants during sample collection
- Exposure to reagents used for sample preservation
- Physical and biological hazards associated with sampling sites specifically when sampling surface waters or wells.

Refer to Chapter 2 of this handbook for specific hazard identification techniques and detailed control methods.

9.6. General Sampling Procedures

General sampling procedures for each common type of drinking water sampling are described in this section.

9.6.1. General Tap Sampling

General tap sampling is appropriate for most drinking water sampling parameters and is the most common drinking water sampling method.

9.6.1.1. Appropriate Parameters

Tap sampling is an acceptable sampling method for the following parameters:

- Inorganic contaminants
- Microbiological
- Metals
- SVOCs
- DPBs
- Radionuclides.

9.6.1.2. Sampling Procedures

The general procedure for collecting samples from a tap includes the following steps:

1. Determine the appropriate sample location and assemble all necessary materials. The proper preservative should be added by the laboratory or be available in the field for addition to the sample. A dechlorinating agent is used when sampling chlorinated waters (such as those found in the distribution system). Handle the sterilized containers carefully to avoid contamination.
2. Go to the sampling location(s) specified in the SAP. Drinking water sampling locations are usually located in the distribution system and are accessible during the day. Examples include hospitals, city buildings, restaurants, pump stations, and dedicated sampling stations. The tap should be clean and in good condition (i.e., no leaks). To minimize contamination, avoid drinking fountains and faucets with swivel necks unless specific point-of-use concerns arise. If the sampling location does not have an accessible tap, then a separate, clean, 1-liter plastic or glass (as appropriate) container should be used to dip and fill the sample bottle(s). Point-of-use filters and leaking taps or faucets that allow water to flow outside of the tap should be avoided.
3. Before turning on the tap, remove any aerator, strainer, or hose to prevent contamination; however, you might not be able to remove the aerator or find a non-swivel faucet. The screen should be removed prior to sampling for bacteria or volatile organics because the screen tends to aerate the water, resulting in the loss of some organics and non-representative samples. The removal of the screen should be documented. When sampling for bacteria, do not take a sample from a swivel faucet, because the joint can harbor a significant bacterial population.
4. Open the cold-water tap, and allow the system to flush to clear the service line from the building to the distribution main. Allow the water temperature to stabilize for 2 – 3 minutes before collecting the sample.
5. Fill out the sample label using waterproof ink. Be sure to clearly identify the exact sample collection location and the date and time of collection. If the sample collection point has a specific coded identification, include it on the label and the COC record along with the sampler's name.
6. Adjust the flow from the tap to about 500 milliliters (1 pint) per minute (approximately ⅛-inch diameter stream).
7. Check for steady flow. Do not change the water flow once sampling has started.
8. Remove the bottle cap or open the plastic bag. Add preservative if required. Position the bottle or bag under the water flow. Hold the bottle in one hand and the cap in the other. When filling a bottle, tip the bottle slightly so that water flows down the side wall of the container. Fill the bottle to the shoulder about ¼ inch from the top of the bottle. For VOCs, fill the bottle just to the point of overflowing. If using a plastic bag sampling container, fill it to the marked fill line. If the preservative is acid, add it after the sample is collected.

WARNING: Do not lay the cap down or put it in a pocket. Do not contaminate the sterile bottle (or bag), touch the inside of the cap with your fingers, or permit the faucet to touch the inside of the bottle or bag. Do not rinse out the bottle or bag before collecting the sample.

9. For chlorinated systems, check the chlorine residual and record; see Section 9.7.1.
10. Place the cap on the bottle and screw it down tightly. If using a plastic bag, pull the wire tabs and whirl the bag three times for a tight seal.
11. Turn the tap off. Replace the aerator, strainer, or hose.
12. Check that the information on the label is correct.
13. Complete the necessary forms with the appropriate information, including the PWS identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, distribution system). Specify the type of analysis to be run. Complete a COC record.

9.6.2. Service Line Sampling

Service line sampling is required if a limited lead service line replacement program is being considered or when the service line material is unknown.

9.6.2.1. Appropriate Parameters

Service line sampling is an acceptable method for sampling metals.

9.6.2.2. Sampling Procedures

The general procedure for collecting samples from a service line is as follows:

1. Determine the appropriate service line sample location and assemble all necessary materials. The proper preservative should be added by the laboratory or available in the field for addition to the sample. Handle the sterilized containers carefully to avoid contamination.
2. Fill out the label for the sampling bottle in waterproof ink. Be sure to clearly identify the exact sample collection location and the date and time of collection. If the sample collection point has a specific coded identification, include it on the label and the COC record along with the sampler's name.
3. Using one of the following three procedures, ensure that service line water is being sampled:
    a) At the tap, flush an appropriate volume of water from the building plumbing before collecting the sample. This removes the water contained in the pipes between the sampling tap and the service lines. Use a container, such as a pitcher or bucket, for measuring the volume of water wasted. Table 9-1 provides an abbreviated list of the volume to be flushed.
    b) For single-family homes, allow the water to run until there is a significant change in its temperature. The temperature change indicates that water is flowing from the service line(s) outside the home.
    c) Locate or install a sampling tap directly on the service line, and sample from this tap.

Table 9-1. Approximate Volume, in Gallons, of Flush Water for Various Sizes and Length of Copper Pipe

Nominal Pipe Size (inches)	Length of Pipe (inches)					
	10	**20**	**30**	**40**	**50**	**100**
½	0.1	0.2	0.3	0.4	0.5	1.0
¾	.23	.16	0.4	0.9	1.1	2.3
1	.41	.82	1.2	1.6	2.0	4.1

4. After step 3a, 3b, or 3c, immediately collect the sample in a 1-liter plastic bottle or collapsible container while the water is still flowing. For 3c, turn on the service line tap, and collect the water immediately. Remove the bottle cap, or open the plastic bag. Add preservative if required. Position the bottle or bag under the water flow. Hold the bottle in one hand and the cap in the other. Fill the bottle to the shoulder, about ¼ inch from the top of the bottle. If using a plastic bag sampling container, fill it to the marked fill line.

WARNING: Do not lay the cap down or put it in a pocket. Do not contaminate the sterile bottle (or bag), touch the cap with your fingers, or permit the faucet to touch the inside of the bottle or bag. Do not rinse out the bottle or bag before collecting the sample.

5. For chlorinated systems, check the chlorine residual and record; see Section 9.7.1.
6. Place the cap on the bottle and screw it down tightly. If using a plastic bag, pull the wire tabs and whirl the bag three times for a tight seal.
7. Turn the tap off.
8. Check that the information on the label is correct.
9. Complete the necessary forms with the appropriate information, including the PWS identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, distribution system). Specify the type of analysis to be run. Complete a COC record.

9.6.3. First-Draw Sampling

First-draw samples are taken to show compliance with ALs. The water in the plumbing must stand motionless in the pipes for at least 6 hours. Taps within a work site building can be secured with tape noting the date, time, and person securing the area. Post signs to warn potential users not to turn on the water during the test period.

9.6.3.1. Appropriate Parameters

First-draw sampling is an acceptable method for sampling metals, particularly copper and lead.

9.6.3.2. Sampling Procedures

The following is a detailed first-draw sampling procedure.

1. All samples for compliance monitoring must be first-draw samples, taken from a kitchen cold water or bathroom cold water sink tap; avoid drinking water fountains. The proper preservative should be added by the laboratory or available in the field for addition to the sample. Handle the sterilized containers carefully to avoid contamination.
2. Fill out the sample label to indicate the date and time of sample collection, the location, type of samples, and the sampler’s name. The site should be sealed, or a certification by the homeowner must be signed to indicate the length of time the system was not used.

CAUTION: Any use of the taps during the 6-hour rest time that the tap is to be secured will invalidate that tap for sampling. Do not turn valves or otherwise mechanically shut down the tap as this can release high amounts of lead and copper into the waterline. Secure taps by taping and posting notices.

3. Before turning the tap water on, remove the cap from the container and position the container under the kitchen or bathroom sink faucet. Turn on the cold water and begin filling the container. Do not put the cap of the container down on any surface. Hold the bottle in one hand and the cap in the other.

WARNING: Do not lay the cap down or put it in a pocket. Do not contaminate the sterile bottle, touch the cap with your fingers, or permit the faucet to touch the inside of the bottle. Do not rinse out the bottle before collecting the sample.

4. Fill the bottle to the shoulder, about ¼ inch from the top of the bottle.
5. Turn off the water tap. Place the cap on the bottle and screw it down tightly.
6. Check that the information on the label is correct.
7. Complete the necessary forms with the appropriate information, including the PWS identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, distribution system). Specify the type of analysis to be run. Complete a COC record.

9.6.4. Source Water Sampling (Surface Water)

Source surface water sampling is appropriate for drinking water parameters because initial contamination will determine the level of treatment required to be in compliance with federal, state and local statutes.

9.6.4.1. Appropriate Parameters

Source water sampling of surface waters is an acceptable sampling method for the following parameters:

- Microbiological
- Metals
- SVOCs
- Inorganic contaminants
- Radionuclides.

9.6.4.2. Sampling Procedures

Refer to Chapter 6, Surface Water Sampling, for further guidance and sampling procedures.

9.6.5. Source Water Sampling From a Well

Source water sampling from wells is appropriate for most drinking water sampling parameters, as it will determine the level of treatment required to bring the water into compliance with federal, state, and local statutes.

9.6.5.1. Appropriate Parameters

Source water sampling of wells is an acceptable method for the following parameters:

- Microbiological
- Metals
- SVOCs
- Inorganic contaminants
- Radionuclides.

9.6.5.2. Sampling Procedures

The general procedure for collecting source water samples from wells is as follows:

Obtain as much information as possible from the well owner, including depth of the well, well yield, formation in which the well is completed, screen depth and length, well construction material, diameter of casing and when and by whom the well was installed. This information should be verified if possible by obtaining drilling logs, etc. With this information, the correct sampling approach, as well as the number of gallons to be evacuated, can be determined so that the collected sample represents the condition of the groundwater.

1. When collecting a sample from an operating domestic well, it is essential to evacuate the plumbing and water storage tank. Running the water for a minimum of 15 minutes before collection is a good rule of thumb; however, a longer evacuation period is desirable. Listen for the pump or the electric circuit to the pump to come on, indicating that the plumbing is being evacuated.
2. Determine if any treatment units are installed on the system. Treatment such as softening, iron removal, turbidity removal, disinfection, and pH adjustment can provide misleading analytical results depending on the parameters of interest. Carbon filters for the removal of organics are also increasingly popular and can alter sampling results. If a treatment system is in use, a basement or outside faucet can bypass such treated water.

Note: Sampling from the cold water faucet is preferred to avoid samples heated by the water heater.

3. A brief inspection of the system should be performed to locate the well, pump, storage tanks, and any treatment systems. Samples should be taken as close to the pumping well as possible and prior to any storage tanks or treatment systems. If a sample must be taken following a treatment unit, the type, size, and purpose of the unit should be noted in the FLB/FN.
4. Faucets, particularly kitchen faucets, usually have a screen installed on the discharge. The screen should be removed prior to sampling for bacteria or volatile organics because the screen tends to aerate the water, resulting in the loss of some organics, resulting in a non-representative sample. The removal of the screen should be noted. When sampling for bacteria, do not take a sample from a swivel faucet because the joint can harbor a significant bacterial population.

Note: A homeowner’s plumbing system should not be tampered with in any way, except for removal of the faucet screen with permission from the homeowner.

For long-term monitoring projects utilizing domestic wells, a specific tap or faucet should be designated as the target sample access point for accurate reproducibility of future samples.

9.7. Parameter-Specific Sampling Procedures

Sampling procedures for regulated and unregulated contaminants are presented in this section by group for the most frequently collected parameters. Sampling procedures for newly regulated contaminants and other parameters of concern can be found in EPA approved methods (http://water.epa.gov/scitech/drinkingwater/labcert/methods_index.cfm)

The parameters and method-specific QC are found in the approved drinking water methods. This section provides common parameter-specific sampling procedures that meet federal requirements. State and local requirements can be different or more stringent; therefore, the sampler should always check state and local sampling requirements before the start of sampling.

9.7.1. Field Data

pH, temperature, chlorine residual, and turbidity measurements are conducted in the field at the start of sample collection.

9.7.1.1. Applicable Regulations

The SDWA requires EPA to institute rules and regulations to ensure that drinking water is safe to consume. The SWTR and subsequent amendments, were promulgated to minimize pathogen contamination in drinking water. These are discussed in Section 9.2. Disinfection and filtration for surface water and groundwater under the influence of surface water is required.

Chlorine residual must be checked to determine preservation, holding time, and, in the case of bacteria testing, data interpretation. Chlorine residual is measured when collecting samples in the drinking water program as free chlorine residual. Total chlorine residual is measured by water supply systems during treatment design, process control studies, or for distribution system problem solving.

MCL or Sampling Values. The regulator should specify the requirements for pH, temperature, and chlorine residual values for compliance. The turbidity after conventional or direct filtration should be ≤ 0.3 nephelometric turbidity units (NTU) in 95% of all monthly samples collected. At no time should the turbidity exceed 1.0 NTU. Slow sand and diatomaceous earth filtration systems must achieve a filtered water turbidity level of ≤ 1 NTU in 95% of the measurements each month (limits can vary by state). At no time can the filtered water turbidity exceed 5 NTU.

9.7.1.2. Number and Frequency of Drinking Water Samples

The pH, temperature, chlorine residual, and turbidity samples should be representative of the system’s filtered water. These measurements should be collected once per day and at the start of each sample collection. Turbidity must be monitored every 4 hours on combined filter effluents, although individual states can reduce this requirement to once-per-day for slow sand or alternative filtration devices. For individual filters, turbidity must be monitored every 15 minutes.

9.7.1.3. Sampling Locations

The regulator should specify where the pH, temperature, and chlorine residual samples must be taken for compliance. One turbidity sample should be taken at each entry point into the distribution system.

9.7.1.4. Analytical Methods

The free chlorine residual method uses the reagent diethylphenylenediamine (DPD) in a colorimetric test to determine the amount of free chlorine available for disinfection. Total chlorine indicates how much of the added chlorine reacted with the indigenous components in the sample and can be determined by adding small amounts of potassium iodide to the DPD solution and recording the difference. The reagent used must be clearly identified as measuring either free chlorine or total chlorine. The results reported must also indicate free or total chlorine measurements.

Nephelometery is a standard test method for turbidity. It is based on comparison of the intensity of light scattered by the contained static water sample under defined conditions to the intensity of light scattered by a reference standard in the sample container. The higher the intensity of scattered light, the higher the turbidity of the sample.

9.7.1.5. Sample Volume

Sample volume depends on the method used for each parameter; refer to Chapter 13 for a discussion of pH, temperature, and chlorine residual field measurements. The turbidity volume collected should be sufficient to:

- Ensure a representative sample
- Allow for replicate analysis (if required)
- Minimize waste disposal
- Comply with the analytical method used.

9.7.1.6. Bottle Type

Bottle type depends on the method used for each parameter; refer to Appendix B for a discussion of pH, temperature, and chlorine residual field measurements. Samples for turbidity should be collected in plastic or glass bottles, as specified by the selected analytical method.

9.7.1.7. Sampling Procedures

The field testing procedures for pH, temperature, and chlorine measurements are presented in Chapter 13. Chlorine residual test kits, including instructions, are available for drinking water testing to measure free or total chlorine. An example of a chlorine residual method can be found in Chapter 13. Most kits use DPD powder, tablets, or drops of liquid placed in a glass tube containing the sample. A color change after adding the reagent indicates that free chlorine is present. In some kits, the color is measured against a field comparator or portable spectrophotometer showing concentration in mg/L or ppm.

If a free residual chlorine level of 0.1 ppm is detected, this test will show as a trace of pink color. Trace colors are visible by looking down the tube and against a white background, which ensures no reflection from any red or pink surrounding surfaces. Interferences in the measurement of chlorine can cause variations to the color or affect the color formation. Any unusual color formations or colors must be noted for proper data interpretation. Note in the FLB/FN the results in mg/L, the date and time of the sampler, the name of the sample, as well as any observations made during the sampling or analysis. If chlorine is not detected in the test, write “not detected.”

A one-time demonstration of technician proficiency should be on file in the training records. The one-time demonstration should include a standard curve, a low-level measurement standard, and an annual performance evaluation sample for chlorine and pH.

For turbidity sampling, all bottles must be thoroughly cleaned and rinsed with turbidity free water prior to sample collection. Turbidity samples are collected typically during general tap sampling, source water sampling from surface water, and source water sampling from wells. Refer to Sections 9.6.1, 9.6.4, and 9.6.5, respectively, for general sample collection procedures.

9.7.1.8. Preservation Requirements

There are no preservation requirements associated with sampling pH, temperature, or chlorine residual. Ensure that samples for turbidity are cooled to ≤ 6°C.

9.7.1.9. Holding Time

Samples for pH, temperature, and chlorine residual must be analyzed immediately, which generally means within 15 minutes of sample collection. Samples for turbidity should be analyzed as soon as possible after collection. If storage is required, samples maintained at ≤ 6°C can be held for up to 48 hours.

9.7.2. Microbiological

This section covers specific sampling requirements for the following microbiologicals:

- Coliforms
- *Cryptosporidium*
- *Giardia*.

9.7.2.1. Coliforms

Coliforms are a group of closely related bacteria that are generally harmless, but do contain some species that are human pathogens. They are natural and common inhabitants of the soil and ambient waters, such as lakes and rivers. Coliforms can be inactivated by treatment or die off naturally in a manner similar to most bacterial pathogens and many viral pathogens. Therefore, if coliforms are found in the distribution system, pathogens also could be present.

9.7.2.2. Applicable Regulations

The TCR introduced in Section 9.2 was established by the SDWA with sample requirements primarily based on the presence or absence of total coliform bacteria rather than on the number of coliforms detected in the samples. The TCR exists to protect public health by determining the presence of disease-causing pathogens in drinking water.

The TCR became effective December 31, 1990 and supersedes the old National Interim Primary Drinking Water Regulations (NIPDWR) for maximum microbiological contaminant levels (effective June 24, 1977). The rule requires that total coliform-positive samples be further tested for fecal coliform or *Escherichia coli* (*E. coli*) and that a set of repeat samples be collected for each total coliform-positive sample. When coliforms are detected, additional routine samples must be collected the following month.

9.7.2.3. MCL

For systems collecting fewer than 40 samples per month, no more than one sample per month is allowed to be positive for total coliform. For systems collecting greater than 40 samples per month, no more than 5% of all monthly samples are allowed to be positive. If a system exceeds the monthly MCL, the public must be notified within 14 days per the requirements established in the Public Notification Rule. If an acute MCL violation occurs (i.e., when any fecal coliform is detected on a repeat sample) the public must be notified within 72 hours.

9.7.2.4. Number and Frequency of Samples

The number of samples that must be taken monthly is based on the population served by the water system. Table 9-2 provides an abbreviated list of minimum monthly monitoring requirements. Many community systems routinely sample more than the minimum to monitor the system's status.

Table 9-2. Microbiological Monitoring Frequency for Routine Sampling in PWSs

Population Served	Minimum Routine Samples/Month*
25 – 1,000	1
1,001 – 2,500	2
2,501 – 3,300	3

***** The regulator can specify a sampling frequency of less than once per month for selected systems. A CWS serving more than 3,300 must refer to the CFR. Contact your state drinking water representative for details.

Compliance with the TCR is based on the presence or absence of total coliform bacteria. If all routine samples test negative (absent) for the presence of total coliforms, no additional testing is required for that monitoring period, regardless of whether sampling is routinely conducted monthly, quarterly, or annually. However, the rule requires that total coliform-positive samples (presence) be further tested for fecal coliform or *E. coli* and that a set of three repeat samples be collected for each total coliform-positive sample monthly, quarterly, and annually. Systems that collect one routine sample or fewer per month must collect a fourth repeat sample. If total coliforms are detected in any routine or repeat sample, the sampler must collect at least five routine samples the next month. Table 9-3 provides an abbreviated list of the required frequency for repeat sampling.

Table 9-3. Monitoring and Repeat Sampling Frequency After a Total Coliform-Positive Routine Sample

# of Routine Samples/Month	# of Repeat Samples	# of Routine Samples Following Month
1/month*	4	5/month
2/month	3	5/month
3/month	3	5/month

* Or fewer.

All repeat samples must be collected within 24 hours of notification of the total coliform-positive result, unless the state waives this requirement.

Each set of repeat samples must include the following:

- One sample at the same tap as the original sample
- One sample within five service connections upstream
- One sample within five service connections downstream.

If a system has only one service connection, the state can authorize the system to collect the required set of repeat samples over 4 days, rather than within 24 hours, after being notified of a total coliform-positive result. Per state discretion, such systems can collect a larger volume repeat sample (or samples) instead of the standard 100-milliliter container (e.g., a single 400-milliliter repeat sample or two 200-milliliter repeat samples in one or more sample containers of any size as long as the total volume collected is at least 400 milliliters). If a system collects more than one routine sample per month, the total volume required would be 300 milliliters.

9.7.2.5. Sample Locations

Samples for the TCR are collected from the distribution system based on the number and frequency requirements.

9.7.2.6. Analytical Methods

The method used for fecal coliform and *E. coli* analysis depends on that used for the total coliform test and must be listed as approved for compliance monitoring. EPA Method 1604 is commonly used for total coliforms.

9.7.2.7. Sample Volume

The sample volume depends on the analytical method used. However, at a minimum, one 100-milliliter sample must be collected per test.

9.7.2.8. Bottle Type

Sample containers for collecting coliform samples must be sterilized, hold at least 120 milliliters (4 ounces) of water, and contain sodium thiosulfate if the sampled water is chlorinated. Bottles can be plastic or glass. The laboratory normally supplies the container. Glass-stoppered bottles must be covered with aluminum foil or char-resistant paper prior to sterilization. The covering on the top ensures that the seal between the stopper and the bottle is not contaminated, thus minimizing the potential for contaminating the sample when it is opened. Some labs furnish a single-service, sterilized, polyethylene bag or bottle containing a sodium thiosulfate tablet. The laboratory should perform sterility checks (one per lot) prior to releasing the containers for collecting samples. The laboratory normally provides instructions to go along with the sample containers it has supplied. Refer to those when provided.

9.7.2.9. Sampling Procedures

The laboratory that supplies the sampling containers might provide instructions for the type of monitoring being performed. Be sure to refer to those instructions when provided. Total coliform samples are collected typically during general tap sampling, source water sampling from surface water, and source water sampling from wells. Refer to Sections 9.6.1, 9.6.4, and 9.6.5, respectively, for general sample collection procedures.

9.7.2.10. Preservation Requirements

Coliform samples must be refrigerated or cooled to ≤10°Cimmediately after sampling.

9.7.2.11. Holding Time

The laboratory must receive and begin analyzing coliform samples within 30 hours of collection. If the laboratory is nearby, refrigerate the samples with ice packs and deliver them directly. Samples analyzed by an off-site laboratory should be shipped on ice via overnight service on the day they were collected.

9.7.2.12. Cryptosporidium and Giardia

Cryptosporidium is a genus of protozoan parasites potentially found in water and other media. The recent taxonomy of the genus *Cryptosporidium* includes two species that use humans as hosts: *C. hominis* and *C. parvum*.

Giardia is a genus of protozoan parasites potentially found in water and other media. The recent taxonomy of the genus *Giardia* includes one species that uses humans as hosts: *G. lamblia* (also called *G. intestinalis* or *G. duodenalis*).

Applicable Regulations. Under the 1996 SDWA Amendments, EPA must supplement the existing 1989 SWTR with additional pathogen control requirements. Several additions to the rule have resulted in the Long-Term 2 Enhanced SWTR, which focuses on the control of *Cryptosporidium* and microbial pathogens.

MCLG. The Interim Enhanced Surface Water Treatment Rule (IESWTR) has an MCLG of zero or a 99% removal for systems that filter.

Number and Frequency of Drinking Water Samples. Monitoring of source water is required to determine the level of treatment for *Cryptosporidium*. Monthly samples are required for the first 2 years, with the level of *Cryptosporidium* determining the level of any subsequent treatment. Most systems will not require additional treatment. A 6-year follow-up sample is required to ensure continued compliance. Small systems can monitor for *E. coli* and will monitor for *Cryptosporidium* only if the *E. coli* concentrations exceed a specified level.

Sampling Locations. Samples are collected from source water for distribution systems.

Analytical Methods. EPA Method 1623 can be used to measure both *Cryptosporidium* and *Giardia*. Prior to analysis, the water sample must be filtered and the extraneous materials retained on the filter. Qualitative analysis is performed by scanning each slide well for objects that meet the size, shape, and fluorescence characteristics of *Cryptosporidium* and *Giardia* oocysts. Quantitative analysis is performed by counting the total number of objects on the slide confirmed as oocysts. Quality is assured through reproducible calibration and testing of the filtration, immunomagnetic separation (IMS), staining, and microscopy systems.

Sample Volume. The sample volume depends on the analytical method used. Generally, 10-liter bulk samples are taken.

Bottle Type. Ten-liter collapsible containers generally are used for collecting *Cryptosporidium* and *Giardia.* These containers must be sterilized, and are normally supplied by the laboratory. The laboratory should perform sterility checks (one per lot) prior to releasing the containers for collecting samples. The laboratory typically provides instructions to accompany the sample containers. Refer to those when provided.

Sampling Procedures. The laboratory that supplies the sampling containers can provide instructions for the type of monitoring being performed. Be sure to refer to those instructions when provided. *Cryptosporidium* and *Giardia* samples are collected typically during general tap sampling, source water sampling from surface water, and source water sampling from wells. Refer to Sections 9.6.1, 9.6.4, and 9.6.5, respectively, for general sample collection procedures.

Preservation Requirements. Ambient water samples are dynamic environments and, depending on sample constituents and environmental conditions, *Cryptosporidium* and *Giardia* oocysts present in a sample can degrade, potentially biasing analytical results. Samples should be chilled to reduce biological activity between collection and analysis.

If samples are collected early in the day, they should be chilled by storing in a refrigerator between 1°C and 10°C or on ice in a cooler. If the sample is pre-iced before shipping, the ice should be replaced with fresh ice immediately before shipment. If samples are collected later in the day, these samples can be chilled overnight in a refrigerator between 1°C and 10°C. This procedure should be considered for bulk water samples that will be shipped off site because it minimizes the potential for water samples collected during the summer to melt the ice in which they are packed and arrive at the laboratory at > 20°C. If samples are shipped after collection at > 20°C with no chilling, the sample will not maintain the temperature necessary to retard microbiological activity and will not present a representative sample for analysis.

PWSs shipping samples to off-site laboratories for analysis should include in the shipping container a means for monitoring the temperature of the sample during shipping to verify that the sample did not freeze or exceed 20°C. PakSense labels adhere to the samples and provide accurate readings of the temperature environment experienced by the samples in shipment.

Holding Time. Sample preparation must be initiated within 96 hours of sample collection (if shipped to the laboratory as a bulk sample) or filtration (if filtered in the field). The laboratory must complete elution, concentration, and purification in one workday. It is critical that these steps be completed in one workday to minimize the time that any target organisms present in the sample sit in eluate or concentrated matrix. This process ends with the application of the purified sample on the slide for drying.

9.7.3. Metals

EPA has determined that metals are health concerns at certain levels of exposure. Young children and pregnant woman are especially at risk from high levels in their blood. Some of the most pronounced effects in children are interference with growth, deficits in IQ, and altered physical and mental development.

Applicable Regulations. The SDWA has established MCLs for various metals (see Table 9-4). The LCR applies to lead and copper and addresses the effects of plumbing materials on the distribution system as well as ways to minimize their concentration.

Table 9-4. Metal MCL or AL

Metal	MCL (mg/L)
Antimony	0.006
Arsenic	0.010
Barium	2
Beryllium	0.004
Cadmium	0.005
Chromium	0.100
Copper	AL= 1.3
Lead	AL= 0.015
Mercury	0.002
Selenium	0.05
Sodium	20
Thallium	0.002

Prior to December 1992, the MCL for lead was 0.05 mg/L. Since then, instead of establishing a new MCL, a treatment technique requirement is triggered by exceeding an AL. The AL for lead is 0.015 mg/L, and the AL for copper is 1.3 mg/L measured at the 90th percentile at the consumer tap. This means that some samples can exceed 0.015 mg/L for lead and 1.3 mg/L for copper and still not exceed the AL. Specific rules for calculating the 90th percentile can be found in 40 CFR 141.80.

Number and Frequency of Samples. All systems must be monitored annually for all regulated metals except for lead and copper. Sodium samples must be collected annually for surface waters, and every 3 years for groundwater sources. In areas where the sodium content is variable, more frequent analyses might be required. All systems must be monitored for lead and copper for a minimum of two consecutive 6-month periods until one of the following occurs.

- Lead or copper concentrations exceed the MCLs or ALs. Then, additional samples from the distribution system and point of entry must be collected for water quality parameters and corrosion control treatment studies performed and reviewed by state personnel.
- Lead and copper concentrations meet the MCLs or ALs for two consecutive periods. Then, the water supply can petition the regulator to reduce the number of tap water

sampling sites and reduce the frequency of collection.

The number of lead and copper samples taken during the 6-month sampling period is based on the population and the results from previous samples. After two 6-month sampling periods of meeting the MCL or AL, a system can request reduced monitoring (see Table 9-5). Monitoring frequency also can be reduced after several years of meeting the AL. Refer to 40 CFR 141 for specific information.

Table 9-5. Number of Samples for Standard and Reduced Monitoring for Lead and Copper

System Size (Population Served)	Number of Sites (Standard Monitoring)	Number of Sites (Reduced Monitoring)
3301 – 10,000	40	20
501 – 3,300	20	10
101 – 500	10	5
up to 100	5	5

Sampling Locations. State-approved sampling locations should be selected if any have been designated. If not, representative sampling locations should be selected. First-draw samples should be collected at cold-water taps in homes and buildings that are at high risk for lead and/or copper contamination. In addition, samples must be obtained from each entry point to the distribution system (EPTDS). For a surface water system, the plant effluent tap might be an appropriate sampling location. For a well discharging directly into a distribution system (also known as an EPTDS), a tap on the well's discharge piping after any treatment would be appropriate.

Analytical Methods. The method used for metal analysis must be listed as approved for compliance monitoring. Inductively coupled plasma-atomic emission spectrometry (ICP-AES, EPA Method 200.8) is a common method used for metal analysis. The analysis involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instruments measure characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized, and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by a photosensitive device. Photocurrents from the photosensitive device are processed and recorded by a computer system.

Mercury can be analyzed using the automated cold vapor technique. The flameless atomic absorption (AA) procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

If additional analytical sensitivity is required for mercury (ng/L), EPA Method 1631, Mercury in Water by Oxidation, Purge and Trap and Cold Vapor Atomic Fluorescence Spectrometry (CVAFS) can be employed. A water sample is chemically treated to produce volatile mercury and separated from the solution by purging with an inert gas. The mercury is collected on a gold trap and subsequently desorbed into a fluorescence spectrometer where the intensity of its fluorescence is measured and compared with the intensity of the standards. At this level of detection, precluding the introduction of contamination is critical; care must be taken to insure that clean/ultra clean sampling, transportation and analysis techniques are used. Guidance on these techniques can be found in *Guidance for Implementation and Use of EPA Method 1631 for the Determination of Low-Level Mercury* (EPA 821-R-01-023) and EPA Method 1631.

Sample Volume. Sample volume is specified by the method or the laboratory. One-liter samples are usually taken.

Bottle Type. Sampling containers for metals can be made of plastic or glass and should be 1-liter in volume. A 1-liter plastic bottle is most commonly used.

Sampling Procedure. There are two types of metal sampling procedures.

1. Service line sampling; see Section 9.6.2.
2. First-draw sampling; see Section 9.6.3.

Preservation Requirements. If the analytical requirements are for total metals, field preservation is recommended. Samples are preserved by adding approximately 3 milliliters of concentrated ultrapure nitric acid to the sample bottle. Be extremely careful when adding the acid. Check the sample pH with litmus paper. Indicate if the sample pH is < 2. If the pH is not < 2, add acid in 1-milliliter increments until the pH is < 2. Record the amount of acid used, the ultimate sample pH, the date and time of preservation, the person performing the preservation, and the source of the acid used. Indicate on the label whether the sample has been acidified. If the analytical requirements are for other than total metals, check with the method for specific preservation techniques and requirements.

WARNING: Concentrated acids and other chemical preservatives represent a safety concern. Refer to the HASP and Chapter 2 for related precautions and PPE.

Holding Time. Samples can be shipped to the laboratory and preserved by the laboratory within 14 days after sample collection. Samples preserved at the laboratory are not to be analyzed until 24 hours after preservation. Preserved samples should be analyzed as soon as possible, but are stable (except mercury) for up to 6 months.

9.7.4. Nitrates/Nitrites.

Nitrate is a form of nitrogen combined with oxygen. It can be converted in the body to nitrite. High concentrations of nitrate can cause serious illness in infants less than 6 months of age. Nitrate, one of the most widespread of contaminants, can get into water if a well is improperly constructed or located where it is subject to sources of contamination.

Applicable Regulations. NPDWRs establish MCLs for nitrates and nitrites.

MCL. The MCL for nitrate is 10 mg/L, and the MCL for nitrite is 1 mg/L.

Number and Frequency of Samples. One sample should be collected from each entry point or from points in the distribution system that represent each source or treatment plant. The frequency of sampling for initial nitrate monitoring is one sample per quarter for surface water and one annual sample for groundwater. If sample results are greater than 50 percent of the MCL, then quarterly monitoring should be performed until four reliable and consistent sample results that are less than 50 percent of the MCL are obtained. The state may allow a reduced sampling frequency for surface water (annually), provided that analytical results from four consecutive quarters are less than 50 percent of the MCL. For groundwater, the state may return sampling frequency to annually after four consecutive quarterly samples are less than 50 percent of the MCL.

Sampling Locations. Drinking water compliance samples for nitrates and nitrites are taken at the entry point to the distribution system after treatment. Surface water supplies can take samples from the distribution system at a point that is representative of each source after treatment.

Analytical Methods. If an ion chromatograph (IC) is available, nitrates can be analyzed by EPA Method 300. In this method, a small volume of sample is introduced into an IC. The anions of interest are separated and measured using a system consisting of a guard column, separator column, suppressor device, and conductivity detector.

Sample Volume. Sample volume is specified by the method or the laboratory. One-liter samples are usually taken

Bottle Type. Both nitrate and nitrite samples should be collected in 1-liter glass or plastic bottles.

Sampling Procedures. Nitrate and nitrite samples typically are collected during general tap sampling, source water sampling from surface water, and source water sampling from wells. Refer to Sections 9.6.1, 9.6.4, and 9.6.5, respectively, for general sample collection procedures.

Preservation Requirement. Samples should be preserved per method requirements. All samples should be packed on ice to lower the temperature to ≤ 6°C.

Holding Time. Nitrate and nitrite samples should be delivered by the sampler or shipped to the laboratory by overnight courier. Results can be reported as nitrate/nitrite, nitrate only, or nitrite only. Samples of non-chlorinated water must be analyzed within 14 days. Nitrate from chlorinated supplies must be analyzed within 28 days. Samples for nitrite must be analyzed within 48 hours of sample collection. When nitrite and nitrate need to be determined separately, the holding time of 48 hours for nitrite must be met.

9.7.5. Pesticides and Synthetic Organic Chemicals

Organic compounds that volatilize at temperatures greater than 150°C and do not decompose are termed semivolatile organic compounds (SVOCs). The synthetic organic chemicals regulated by the SDWA are SVOCs that fall into the categories of polychlorinated biphenyls (PCBs), pesticides, fungicides, and herbicides. These compounds are commercially produced with applications for agriculture and home use. If incorporated into the water supply, they could impart significant health risks and are consequently regulated under the SDWA.

Applicable Regulations. Since the establishment of the SDWA Amendments of 1986, EPA has established MCLs for PCBs, 23 pesticides, and six other synthetic organic chemicals.

MCLs. A list of drinking water MCLs can be found on the EPA's website, http://water.epa.gov/drink/contaminants/index.cfm.

Number and Frequency of Samples. For systems serving about 50,000 customers and operating within the MCLs, one sample per year is required. For systems operating above the MCLs, quarterly samples are required. The number of samples required and frequency of sampling varies with system size. Consult 40 CFR 141 or http://www.epa.gov/safewater for additional details.

Sampling Locations. State-approved sampling locations should be selected if any have been designated. If none have been designated, representative sampling locations should be selected. Samples are collected after treatment at the treatment plant, or at the entry point to the distribution system, making sure each individual source of water is represented in the sample.

Analytical Method(s). The EPA 500 series methods are designed to identify and quantify organic compounds in municipal drinking water. These methods are cited under the SDWA.

Sample Volume. Sample volume will vary with the parameter being tested. Generally two 1-liter samples are collected.

Bottle Type. All methods require the sample to be collected in glass bottles with Teflon®-lined lids. The size and number of containers will vary depending on the number of parameters to be monitored. In most cases, samples are collected in two 1-liter amber-glass bottles and preserved. Exceptions exist and the methods must be reviewed prior to sampling to ensure the proper size, number, and types of containers are selected.

Sampling Procedures. Samples for pesticides and synthetic organic chemicals are collected typically during general tap sampling, source water sampling from surface water, and source water sampling from wells. Refer to Sections 9.6.1, 9.6.4, and 9.6.5, respectively, for general sample collection procedures.

Preservation Requirement. The preservatives used vary in concentration, addition, and type, depending on the method, parameters to be measured, and the matrix (refer to 40 CFR 141). All samples should be cooled to ≤6°C during transportation and storage. Chlorinated and non-chlorinated water supplies are preserved using different preservatives. Preservatives include sodium thiosulfate to remove chlorine, HCl to stabilize the pH and reduce biological activity, and other chemicals such as mercuric chloride. The use of mercuric chloride requires that the samples and waste material be treated as a hazardous waste for mercury.

9.7.6. DBPs

Chlorine and other chemical disinfectants have been widely used by PWSs as a principal barrier to microbial contaminants in drinking water. DBPs are formed when disinfectants interact with organic and inorganic materials in source waters.

The levels of DBPs in finished water also are dependent on the pH, temperature, concentration of precursor materials, and age of the water in the distribution system. Drinking water can vary significantly from one point in a distribution system to another. The regulated DBPs included in this section are:

- Bromate
- Chlorite
- HAA5
- Total trihalomethanes (TTHMs).

Applicable Regulations. DBPs are regulated through the Stage 1 and Stage 2 of the DDBPR. The Stage 1 DDBPR applies to water systems that add a chemical disinfectant. The Stage 2 DDBPR, finalized in January 2006, makes incremental changes to the Stage 1 rule for additional protection of public health.

The Stage 2 DDBPR is designed to protect public health by supplementing existing drinking water regulations with risk-targeted monitoring and compliance determinations for MCLs. Under the Stage 2 DDBPR, a water system will conduct an evaluation of its distribution system to identify the locations with high DBP concentrations. These locations will then be used as the sampling sites for DDBPR compliance monitoring. Monitoring is required for populations of over 10,000.

MCL. The MCL for each disinfection byproduct covered in this section can be found in Table 9-6.

Table 9-6. DBPs/MCLs

Disinfection Byproduct	MCL (mg/L)
Bromate	0.010
Chlorite	1.0
HAA5	0.060
TTHMs	0.080

Number and Frequency of Drinking Water Samples. The number of samples, frequency, and location of monitoring is dependent on source water, distribution size, initial monitoring results, and state requirements. In general, sampling is required annually for small ground water systems and small subpart H systems. Sampling is required once per quarter for all other systems.

Sampling Locations. Samples are collected within the distribution system after treatment.

Analytical Methods. Ion chromatography by EPA Method 300 is a common method used for the analysis of the inorganic fraction of DBPs. A small sample volume is introduced into an IC. The anions of interest are separated and measured using a system consisting of a guard column, analytical column, suppressor device, and conductivity detector. The organic DBPs are extracted and analyzed using gas chromatography (GC) or GC/MS techniques. EPA Methods 501 and 524 are commonly used for TTHMs, and EPA Method 552 is commonly used for HAA5.

Sample Volume. Sample volume is specified by the method or the laboratory. Two 40- or 60-milliliter samples are usually taken

Bottle Type. The laboratory normally supplies the containers, preservatives, and blank water for DBP sampling. Recommended containers are glass, 60- to 120-milliliter screw-cap vials with a Teflon®-faced silicone septum. The preservatives, containers, and blank water should be traceable and controlled to ensure no detectable DBPs are present.

Sampling Procedures. DPB samples are collected typically during general tap sampling, source water sampling from surface water, and source water sampling from wells. Refer to

Sections 9.6.1, 9.6.4, and 9.6.5, respectively, for general sample collection procedures. Collect all samples in duplicate, and prepare two field blanks for each sampling site. Collect samples as follows:

1. Hold the vial at an angle, and fill it as near to the top as possible without overflowing. Be careful not to rinse out the preservative. No air bubbles should pass through the sample as the bottle is filled.

WARNING: Do not lay the cap down or put it in a pocket. Do not contaminate the sterile bottle (or bag), touch the cap with your fingers, or permit the faucet to touch the inside of the bottle or bag. Do not rinse out the bottle or bag before collecting the sample.

2. When sampling from an open body of water, fill a clean, empty (no preservative), 1-quart, wide-mouth bottle with sample and carefully fill two sample vials from the wide-mouth bottle. Discard the water in the wide-mouth bottle after filling the vials.
3. If a preservative is to be added, do not completely fill the vial until after the preservative has been added. After the preservative is added, carefully fill the bottle.
4. When filling the bottle, form a meniscus (the curved upper surface of a liquid formed by surface tension) or gently pour sample water from the vial cap to top off the bottle and form a meniscus.
5. Screw the cap on the bottle so that the shiny, Teflon® side of the septum is in contact with the water. Do not touch the septum, and do not over-tighten.
6. Invert the bottle, tap against your other hand, and check for air bubbles. If any are present, add additional water to reform the meniscus and check again until no air bubbles are present.

WARNING: Air bubbles larger than approximately 1 millimeter can invalidate the sample. Glass vials must be filled completely and handled with care to prevent spillage.

Preservation Requirement. For collecting chlorinated samples, a dechlorinating agent such as 25 milligrams ascorbic acid should be pre-added by the laboratory supplying the sample containers. If gases are not to be determined, sodium thiosulfate can be used as the dechlorinating agent. It should rapidly dissolve as the bottle is filled. For samples that require HCl, add one drop of HCl to each 20 milliliters of sample volume. Add the water before the HCl to ensure that the HCl and sodium thiosulfate do not react to form sulfur dioxide, which can affect the laboratory results. Sample pH should be < 2 after adjustment. Table 9-7 gives a summary of recommended preservatives for different VOCs.

WARNING: Concentrated acids and other chemical preservatives represent a safety concern. Refer to the HASP and Chapter 2 for related precautions and PPE.

Table 9-7. Recommended Preservatives for VOCs

Constituents	Chlorinated	Non-chlorinated
Halocarbons & Aromatics	HCl + reducing agent	HCl
THMs	Reducing agent (HCl optional)	None required

Holding Time. Pack the samples and blanks with ice to lower the temperature to ≤6°C. Ship to the laboratory by overnight courier or deliver directly. Refer to method holding time requirements.

9.7.7. Radionuclides

The EPA sampling requirement for radionuclides is intended to improve public health protection by reducing exposure to, and the related cancer risk posed by, radionuclides.

Applicable Regulations. Since December 2003, the Radionuclide Rule has included the existing MCLs for combined radium-226 and radium-228, gross alpha particle radioactivity, and beta particle and photon activity. The rule also established a new MCL for uranium. It is important to note that the Radionuclide Rule regulates CWSs only.

MCL. The MCL for each regulated radionuclide can be found in Table 9-8.

Table 9-8. Radionuclide MCL

Radionuclide	MCL
Alpha particles	15 picocuries per liter (pCi/L)
Beta particles and photon emitters	4 millirems per year
Radium-226 and radium-228	5 pCi/L
Uranium	30 µg/L

Number and Frequency of Drinking Water Samples. Samples for radionuclides are collected once every 4 years unless more frequent sampling is required by the state. Initial monitoring is performed by collecting one sample per quarter for 1 year. Repeat monitoring can be reduced to a single grab sample, if approved by the state. If gross alpha particle activity is found to be above the MCL, additional monitoring for radium-226 and radium-228 might be required. If the water source is near nuclear facilities, additional parameters (e.g., tritium, strontium-89, strontium-90, iodine-131, and cesium-134) might need to be monitored. These additional parameters will be specified in the state or federal permit.

Sampling Locations. State-approved sampling locations should be selected if any have been designated. If not, representative sampling locations should be selected. For a surface water system, the plant effluent tap might be an appropriate sampling location. For a well discharging directly into a distribution system (also known as an EPTDS), a tap on the well's discharge piping after any treatment would be appropriate.

Analytical Methods. Alpha particles are measured by reducing the sample using evaporation or a suitable chemical method to the minimum weight of material having measurable alpha activity. Alpha radioactivity is measured by an instrument composed of a detecting device, amplifier, power supply, and scaler– the most widely used being proportional, and scintillation counters. Gross alpha can be measured using EPA Method 900.

Beta radioactivity can be measured by one of several types of instruments composed of a detecting device and combined amplifier, power supply, and scaler– the most widely used being proportional or Geiger-Muller counters. Where a wide range of counting rates is encountered (0.1 – 1,300 counts per second), the proportional-type counter is preferable due to a shorter resolving time and greater stability of the instrument. The test sample is reduced to the minimum weight of solid material having measurable beta activity by precipitation, ion exchange resin, or evaporation techniques. Beta particles entering the sensitive region of the detector produce ionization of the counting gas. The negative ion of the original ion pair is accelerated towards the anode, producing additional ionization of the counting gas and developing a voltage pulse at the anode. By use of a suitable electronic apparatus, the pulse is amplified to a voltage sufficient for operation of the counter scaler. The number of pulses per unit of time is related to the disintegration rate of the test sample. Gross beta radiation also can be measured using EPA Method 900.

Radium-226 and radium-228 are measured by using sequential precipitation and an internal proportional counter or scintillation counter. Radium-226 and radium-228 in water are concentrated and separated by coprecipitation with sulfates of lead and barium and purified by ethylenediaminetetraacetic acid (EDTA) chelation. After 36-hour ingrowth of actinium-228 from radium-228, actinium-228 is carried on yttrium oxalate, purified, and beta-counted. Radium-226 in supernatant is precipitated as sulfate, purified, and alpha-counted (per 7500-Ra B), or is transferred to a radon bubbler and determined by an emanation procedure (per 7500-Ra C; the preferred method).

Solvent extraction technology is a common method for isolating and concentrating uranium in drinking water by counting via a high-resolution alpha-liquid-scintillation spectrometer. To determine total uranium, as well as limited isotopic uranium (^{238}U and ^{234}U) by activity in drinking water, a 200-milliliter acidified water sample is first spiked with ^{232}U as an isotopic tracer, boiled briefly to remove radon, and evaporated until less than 50 milliliters remain. The solution is then made approximately 0.01 M in diethylenetriaminepentaacetic acid (DTPA), and the pH is adjusted to between 2.5 and 3.0. The sample is transferred to a separatory funnel and equilibrated with 1.50 milliliters of an extractive scintillator containing a dialkyl phosphoric acid extracting agent. Under these conditions, only uranium is quantitatively transferred to the organic phase while the extraction of undesired ions is masked by the presence of DTPA. Following phase separation, 1.00 milliliters of the organic phase is sparged with dry argon gas to remove oxygen, a chemical quench agent, and counted on a high-resolution alpha-liquid-scintillation spectrometer and multichannel analyzer (MCA). Radium-226 can be measured using EPA Method 903, and radium-228 can be measured using EPA Method 904.

Sample Volume. Sample volumes will vary depending on the analytical requirement. Generally, 1 gallon is collected per sample. Sample volumes being collected must be confirmed with the laboratory performing the analysis.

Bottle Type. Containers suitable for radionuclide sampling are either a one pre-cleaned 1-gallon Nalgene plastic bottle, or two pre-cleaned 2-liter plastic bottles per sample.

Sampling Procedures. Radionuclide samples typically are collected during general tap sampling source water, sampling from surface water, and source water sampling from wells. Refer to Sections 9.6.1, 9.6.4 and 9.6.5, respectively, for general sample collection procedures.

Preservation Requirement. Preservation for radionuclides is based on the method, parameter, and state requirements. Cesium-134 is preserved with HCl to pH < 2. Tritium and iodine-131 are not chemically preserved. All other regulated radionuclide parameters are preserved with nitric acid to pH < 2. If preservation is not performed in the field, the preservative can be added upon arrival at the laboratory. The preservative must be added within 3 days of collection, and analysis must not begin until 16 hours after acidification. Preservatives must be checked for the absence of the radionuclides being monitored.

Holding Time. Samples must be delivered to the analytical laboratory within 3 days for proper testing.

9.8. Sampling Equipment List

Section 3.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides specific equipment applicable to drinking water sampling.

- Chlorine
    - Chlorine test kit or DPD test kit
- Microbiological
    - 120 milliliters plastic or glass containers (sterilized)
    - Sodium thiosulfate – a preservative
    - 10-liter collapsible containers
- Metals
    - First Draw
        - 1-liter bottle, plastic or glass
        - Nitric acid – a preservative
        - Warning signs to notify potential users not to turn on taps during testing period
    - Service Line Samples
        - Pitcher or bucket
        - Sampling tap
        - 1-liter bottle, plastic or glass
        - Nitric acid – a preservative
- Synthetic Organic Chemicals
    - Sulfuric acid (for nitrate non-chlorinated water only)
    - Sodium thiosulfate – a dechlorinating agent
    - HCl – a preservative
    - Mercuric chloride – a preservative
    - 1-liter amber glass bottles with Teflon®-lined lids

Note: Samples will be considered a hazardous material once mercuric chloride is added.

- DBPs
    - 40-milliliter amber vials with Teflon®-faced silicone septa
    - Ascobic acid – a dechlorinating agent
    - HCl – a preservative if sampling for halocarbons or aromatics
    - 1-quart wide-mouth bottle

- Radionuclides
  - HCl – a preservative if sampling for Cesium-134
  - Nitric acid – a preservative for all other radionuclides except for tritium and iodine-131
  - 1-gallon or two 1-liter plastic or glass bottles, volume dependent upon sample.

Chapter 10. Waste Sampling

10.1. Purpose

This chapter provides general procedural guidance to technical personnel who sample underground storage tanks (UST), drums, cylinders, articles containing PCBs, investigation derived waste (IDW), and other sources of solid or hazardous wastes that pose a potential threat to human health and the environment. Waste sampling may be performed for site assessments or for meeting regulatory requirements mandating proper storage, handling, and disposal of materials in drums, cylinders, USTs, articles containing PCBs, oil spills, and other solid wastes.

10.2. Scope

The overall objectives of waste sampling are to determine waste composition and meet regulatory monitoring requirements. Waste sampling activities need to be conducted properly to protect the health of those carrying out the sampling and ensure the accuracy and reliability of the sampling results. Waste sampling oversight should be maintained by following a previously prepared SAP; see Chapter 2 for more information. The sampling procedures, documentation, packaging, handling, and transportation requirements contained in the SAP should be followed. Refer to Chapter 3 for a complete discussion of sampling preparation and sample collection requirements applicable to all types of sampling programs.

The following sampling subjects are included within the scope of this chapter:

- USTs
- PCBs
- Containers and drums
- Oil spills
- Waste characterization
- Surface sampling
- Sewage sludge.

WARNING: Sampling personnel should be specifically trained, qualified, and certified to perform waste sampling operations due to the potential for encountering extreme safety hazards during these operations.

Specific objectives of waste sampling, as addressed in this chapter, are as follows:

- Provide relevant data for the protection of personnel
- Determine the presence of any USTs, drums, cylinders, articles containing PCBs, or other solid or hazardous wastes that require special handling
- Determine waste characteristics such as toxicity and physical hazards from reference or known sources
- Determine alternative disposal criteria in accordance with federal, state, and local regulations
- Determine special procedural requirements for the handling and sampling of PCB-contaminated articles, containers, and transformers
- Evaluate the SPCC plan (along with proper health and safety procedures) to ensure that spill sampling requirements are satisfied subsequent to an accidental release from the site
- Define practices and procedures for safe handling of drums and cylinders containing solid or hazardous materials or wastes
- Promote the safe handling of known hazardous materials.

Note: Personnel should follow special precautionary measures with unlabeled drums, cylinders, USTs, articles containing PCBs, and other wastes unless their contents are characterized or known.

10.2.1. USTs

UST sampling provides information used to determine compliance with regulations applicable to USTs that contain petroleum products and stored chemical substances. If locating the USTs is an issue, then an electromagnetic wave device, electrical resistivity meter, or ground-penetrating radar can be used to estimate the location of buried tanks.

The following references contain guidance on UST sampling:

- **40 CFR 112.** EPA Regulation on Oil Pollution Prevention
- **40 CFR 280.** EPA Technical Standards and Corrective Action Requirements for Owners and Operators of USTs.

These references cover the following situations:

- When tank volume, including piping, is 10 percent or more beneath the ground surface
- When a tank holds RCRA hazardous wastes, or a mixture of such wastes and other regulated substances.

These federal regulations also outline procedures for EPA approval of a state program to operate in place of federal UST requirements. State standards must be no less stringent and must provide adequate enforcement of compliance with these standards. The sampling of USTs should comply with all applicable federal, state, and local regulations pertaining to the management of listed USTs. Sampling event planners should identify the regulatory requirements (including the compliance of each UST) for sampling, monitoring, and removal, where necessary.

10.2.2. PCBs

PCB sampling provides information and guidance for PCB handling in accordance with applicable federal, state, and local regulations.

The following references provide information on PCB sampling, access, and storage:

- **TSCA.** PCB requirements, as implemented by EPA regulations
- **40 CFR 761.** EPA regulations on PCB Manufacturing, Processing, Distribution in Commerce, and Use of PCBs and PCB Items.

Some regulatory requirements from the previously listed references are as follows:

- Areas contaminated with PCBs in regulated amounts must be closed to the public
- All PCB transformers must be registered with the appropriate fire department and labeled per EPA requirements
- PCB storage areas and transport vehicles should be marked with special labels per applicable DOT regulations
- Non-leaking PCB articles and equipment, or leaking PCB items placed in non-leaking containers, may be put in temporary storage areas for up to 30 days
- The site-specific SPCC plan should include PCB sampling and follow proper health and safety procedures for sampling personnel
- PCB transformers or PCB-contaminated materials must be moved to a permanent storage facility if they have been stored in a temporary facility for more than 30 days
- PCB waste is subject to a 1-year time limit for storage and disposal. Conditional extensions can increase this limit by an additional year (40 CFR 761.65)
- Post-cleanup sampling should be performed to verify conformance with cleanup standards (40 CFR 761.125).

When performing field sampling for PCBs, proper health and safety protocol must be followed in accordance with OSHA 29 CFR 1910.120. All items containing PCBs, or unknown items potentially related to PCBs, should be considered potentially contaminated with PCBs and handled according to regulations unless sampling results prove otherwise.

10.2.3. Containers and Drums

Container and drum sampling provides information and guidance applicable to the regulation of container and drum handling in accordance with federal, state, and local regulations. Container and drum sampling poses unique obstacles to sampling personnel, including drum staging, identification, and opening. Containers include compressed gas cylinders.

The condition and content of drums will determine the appropriate procedures for handling drums prior to sampling. All drums should be visually inspected to gain as much information as possible about their content. In most compliance cases, the materials contained in the drum are known. Appropriate procedures for handling the suspected materials should be followed. However, depending on site control of the materials placed in drums, additional safety practices should be considered.

Prior to drum sampling, a survey can be made to classify the drums into preliminary hazard categories, such as the following:

- Chemical incompatibility
- Leaking or deteriorated
- Bulging
- Explosive or shock sensitive
- Laboratory waste.

As a precautionary measure, all unknown drums should be handled and sampled with a minimum of Level B protection (per 29 CFR 1910.120) to characterize the content of the drums. The drum survey should also address unstacked and unorganized drums.

WARNING: Opening drums presents a major health and safety hazard. Accidents may include detonation, explosion, fire, vapor generation, and physical injuries. Proper precautionary measures must be taken while performing drum sampling.

The following references provide information on drum/container sampling:

- **OSHA 29 CFR 1910 and 1926.** General Safety and Health Standards and Safety and Health Regulations for Construction
- **40 CFR 265.** National Standards for the Management of Hazardous Waste
- **49 CFR 171–178.** Established regulations for the Safe and Secure Transportation of Hazardous Materials in Commerce.

10.2.4. Oil Spills

Oil spill sampling offshore and ashore includes the methodology to collect samples following accidental discharge of oil from the site. Ashore oil spill sampling is typically oil sampling at the beach but should not be limited to only surface sampling of the oil. There are two portions of ashore oil spills that are important to sample: 1) the surface layer (0 – 15 centimeters), which reflects the accumulation of the oil from an offshore spill following washout to the beach, and 2) long-term deposition of the contaminated products several feet deep at the beach.

The following references provide information on oil spill sampling:

- **40 CFR 109.** EPA Regulation on Criteria for State, Local and Regional Oil Removal Contingency Plans
- **40 CFR 110.** EPA Regulation on Discharge of Oil
- **40 CFR 300.** EPA Regulation on National Oil and Hazardous Substances Pollution Contingency Plan under CERCLA.

Oil spill sampling includes sampling the spill first. Because each spill and spill site is unique, it is impossible to establish step-by-step guidelines for oil spill sampling. Spill samples should be collected as soon as possible, gathering the greatest volume of oil before it spreads out or disperses. This technique also minimizes the effects of weathering alteration on the spilled oil. Offshore and ashore oil spill sampling should be in concurrence with the SPCC plan and the site-specific HASP. The HASP provides technical guidance associated with collection of samples.

10.2.5. Solid and Hazardous Waste Characterization

Waste sampling provides the identification of waste present, characterizes the waste, and aids in the determination of adverse effects to human health and the environment. This information ultimately supports the selection of disposal options available in accordance with federal, state, and local regulations. RCRA, an amendment to the SWDA to ensure that solid wastes are managed in an environmentally sound manner, provides the following two distinct, yet interrelated, programs:

- **Subtitle D**. Promotes and encourages the environmentally sound management of solid waste
- **Subtitle C.** Promotes and encourages the environmentally sound management of hazardous waste.

10.2.5.1. Characteristics of Hazardous Waste

Characteristic hazardous wastes exhibit any of the following:

- Ignitability
- Corrosivity
- Reactivity

- Toxicity.

10.2.5.2. Ignitability

Ignitable hazardous wastes include the following:

- Liquids (other than aqueous solutions) containing less than 24% alcohol by volume with a flash point of less than 60°C (140°F)
- Non-liquids capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes and, when ignited, of burning to create a hazard
- Ignitable compressed gases
- Oxidizers.

10.2.5.3. Corrosivity

Corrosive hazardous wastes include the following:

- Aqueous solutions having a pH of ≤ 2 or ≥ 12.5
- Liquids that corrode carbon steel at a rate > 6.35 millimeters (0.250 inch) per year.

10.2.5.4. Reactivity

Reactive hazardous wastes have any of the following attributes:

- Cause explosions, toxic fumes, gases, or vapors when heated compressed, or mixed with water
- Are normally unstable and readily undergo violent change without detonating
- Are cyanide- or sulfide-bearing wastes
- Are capable of detonation or explosive reaction when subjected to a strong initiating source or heated under confinement
- Are readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure
- Are listed as forbidden Class A or Class B explosives.

10.2.5.5. Toxicity

A solid waste exhibits the characteristic of toxicity when the extract from a representative sample of the waste contains any of the contaminants listed in Table 1 of 40 CFR 261.24 at the concentration equal to or greater than the respective value given in that table.

10.2.5.6. Listed Hazardous Wastes

Hazardous wastes are also listed in 40 CFR 261.31 – 34. These lists contain industry and EPA hazardous waste numbers, descriptions, and hazard codes (indicating type of hazard). “Non-specific source” wastes such as degreasing solvents are assigned EPA hazardous waste designation numbers beginning with “F.” Wastes that contain pure or commercial-grade unused chemical solvents are assigned EPA hazardous waste designation numbers beginning with “P” or “U;” commercial chemicals on these lists are regulated as hazardous wastes when discarded. Note that “K-list” wastes (hazardous wastes from specific sources) involve in-process wastes and generally are not applicable to this discussion.

10.2.5.7. Characterization by Sampling

Characterizing waste by sampling can be quite expensive. It is preferable to identify wastes by determining the origin of the waste. Once the origin of the waste has been identified, knowledge of the process used to generate the wastes (e.g., raw material consumption, products, and by-products) can be used to narrow the list of analytical methods required for characterizing wastes. For containerized wastes, identifying labels or reviewing paperwork associated with the containers may also narrow the list of analytical methods required. Associated paperwork may include waste manifests, disposal logs, and MSDSs. By reducing the number of analytical tests required, the sampling costs, sample volumes, and resulting waste are reduced.

Field analytical techniques have made significant improvements in rigor and credibility. Computerization and miniaturization are contributing to technology improvements and innovations. These improvements allow for more frequent use of field testing to characterize wastes rather than collecting samples and sending them to a laboratory for analysis. Field analytical methods may be employed if sufficient documentation can be provided to support the proper application of the method. The selected field analysis method should demonstrate method quantitation limits below the action level or levels of concern. Field analysis allows for streamlining of the site investigation process.

10.2.6. Sewage Sludge

Sewage sludge sampling includes collecting and analyzing representative samples of sewage sludge prior to land application, surface disposal, or firing in a sewage sludge incinerator. Sewage sludge sampling also includes sampling of domestic sewage prior to disposal. Sampling data allow for the characterization of sewage sludge to determine disposal methods in accordance with federal, state, and local regulations. The CWA and RCRA are examples of two applicable federal regulations. The CWA regulates beneficial use and disposal of sewage sludge, and RCRA regulates disposal of non-hazardous sludge in a municipal landfill and land disposal of hazardous sludge.

Guidance for sewage sludge sampling is contained in the following references:

- **40 CFR 258.** EPA Regulation on Criteria for Municipal Solid Waste Landfills
- **40 CFR 268.** EPA Regulation on Land Disposal Restrictions
- **40 CFR 503.** EPA Regulation on Standards for the Use or Disposal of Sewage Sludge.

10.2.7. IDW

IDW is waste that is generated in the process of investigating contaminated or potentially contaminated sites. IDW can include purge water, cuttings from well installation, drilling mud, soil from collection of samples, and contaminated PPE. IDW should be sampled and disposed of in accordance with the SAP, contractual requirements, and state and federal requirements. IDW management has the potential for considerable effort and expense, and should be considered when planning an investigation. IDW will generally be sampled using the techniques described in section 10.5 of this chapter.

For more information on managing IDW, consult the EPA's *Guide to Management of Investigative-Derived Wastes* (http://www.epa.gov/superfund/policy/remedy/pdfs/93-45303fs-s.pdf).

10.3. Hazard and Safety Precautions

See Chapter 2 of this handbook for a discussion of health and safety elements applicable to most sampling programs. The following hazards, discussed below, are specific to waste sampling:

- Container hazards
- USTs and other confined spaces
- Explosive hazards.

10.3.1. Container Hazards

Reseal open bungs and drill openings as soon as possible with new bungs or plugs to avoid vapor release. If an open drum cannot be resealed, place it into an overpack drum.

Bulging drums are extremely hazardous. Whenever possible, do not move drums that may be under internal pressure, as evidenced by bulging or swelling.

Laboratory packs (categorizing small containers of chemicals, solvents, industrial supplies, etc. in metal drums for disposal) can be an ignition source for fires at hazardous waste sites, as they sometimes contain shock-sensitive materials. Treat such containers as explosive materials, and use extreme care when opening these packs. Prior to opening laboratory packs, make sure all non-essential personnel are moved to a safe place. Whenever possible, use a grappler unit constructed for explosives containment for initial handling or sampling of such laboratory packs. Sampling personnel should maintain continuous communication with the Site Safety Officer until operations are complete.

WARNING: If drums or cylinders show signs of swelling or bulging, do not move or sample them until the pressure can be relieved safely. If possible, relieve any excess pressure prior to opening the drum by using a remotely controlled device, such as a pneumatic impact wrench or hydraulic penetration device. If pressure must be relieved manually, place explosion-resistant plastic barriers between the worker and the drums or cylinders.

10.3.2. USTs and Other Confined Spaces

For USTs and other confined spaces, the HASP should be followed to ensure worker safety.

WARNING: Personnel who enter a confined space should be accompanied by an on-site standby unit at all times.

The following steps should be detailed in the HASP prior to entry into a confined space:

1. Ventilate the area thoroughly prior to entry.
2. Disconnect connecting pipelines.
3. Take air samples to verify the absence of flammable or other hazardous vapors and demonstrate the presence of adequate oxygen levels.
4. Provide entry and standby teams with proper respiratory protection, protective clothing, safety harnesses, and ropes.
5. Establish lifeline signals (e.g., tugging on a rope) prior to entry so that field sampling personnel can communicate.
6. Maintain ladder(s) and other equipment in accordance with OSHA regulations.

10.3.3. Explosive Hazards

Due to the inherent dangers in sampling explosives (as well as potential or suspected explosives), only those individuals who have been trained and certified in the proper handling of these materials should participate in sampling activities. See OPNAVINST 8023.24B for qualifications and certification requirements. Special considerations are required when sampling explosive wastes that may be susceptible to shock, friction, electromagnetic radiation (EMR), electrostatic discharge, sparks, flames, elevated or freezing temperatures, moisture, or sunlight. Failure to handle explosives correctly could result in damage to property, injury, or loss of life.

General safety considerations include wearing PPE such as flameproof clothing, caps, safety goggles or face shields, conductive shoes, and respirators, if applicable. Only non-sparking tools should be used. Electrical grounding may be necessary in some cases. In addition, field sampling personnel should collect the smallest amount of sample necessary for the intended testing and analysis.

WARNING: Only trained and certified explosives personnel should handle, sample, or test explosives or suspected explosive materials.

10.4. Preparation

If the nature of the hazardous waste is unknown (e.g., at an abandoned disposal site), sampling personnel should take additional precautions to protect themselves from direct contact with waste materials. To characterize waste materials for RCRA or to verify the contents in drums, cylinders, tanks, or transformers, it is important that representative samples be collected and analyzed.

10.4.1. Preparation for UST Sampling

Before starting UST sampling, follow the hazard and safety precautions specified in Section 10.3. Prior to UST sampling, field personnel should ensure there is no leakage of product from the UST or related piping. If it is necessary to enter into a tank or vault for sampling, strict procedures for confined space entry should be followed by qualified and experienced personnel.

10.4.2. Preparation for PCB Sampling

The toxic nature and degree of hazards posed by PCBs require a high level of caution to be taken during sampling. Appropriate protective gear and clothing should be worn by sampling and support personnel during sampling events. Proper gloves should be used during sampling. Spill prevention and control should be planned in advance if transformer sampling is to be performed. Plastic pads and sorbent materials should be available.

10.4.3. Preparation for Drum Sampling

Prior to initiation of a sampling event, all drums should be inventoried. All available information concerning each drum should be recorded in the FLB/FN, including the following:

- Type of drum
- Contents
- Total capacity estimate
- Markings
- Labels
- Color
- Origin
- Condition.

Each drum should be marked with a unique identification number that corresponds to the sample container for present and future reference. Enamel spray paint is suitable for this purpose. Photographs of the numbered drum are useful for documenting the condition of the drum and providing a permanent record.

The opening of closed drums prior to sampling entails considerable risk if not performed with the proper techniques, tools, and safety equipment. Prior to sampling, all drums should be staged in easy-access areas. Any standing water or other materials should be removed from the top of the container so that:

1. The representative nature of the sample is not compromised when the container is opened.
2. Water cannot enter drums containing water-reactive compounds.

Manual and remote drum sampling techniques are discussed below.

WARNING: Before opening any drum, ensure that the drum is in an area that has secondary containment.

10.4.3.1. Manual Drum Opening

Two tools, discussed below, are typically used to open drums manually, bung wrenches and drum deheaders.

10.4.3.1.1. Bung Wrench

The most common method for manually opening drums is with a universal bung wrench. These wrenches have fittings to remove nearly all commonly encountered bungs and are constructed usually of cast iron, brass, or a bronze-beryllium non-sparking alloy formulated to reduce the likelihood of sparks. Use of a non-sparking wrench is highly recommended. However, use of a non-sparking wrench does not completely eliminate the possibility of producing sparks.

The wrench fitting matching the bung is inserted into the bung, and the tool is turned counter-clockwise to remove the bung. Proper protection and safety is necessary while opening drums.

WARNING: If drums or cylinders show signs of swelling or bulging, do not move or sample them until the pressure can be relieved safely.

10.4.3.1.2. Drum Deheader

A drum deheader can be used to open a drum manually when the bung is not removable with a bung wrench. Drum deheaders are constructed of forged steel with an alloy steel blade designed to cut the lid off (or partway off) with a scissor-like cutting action.

Drums are opened with a drum deheader by first positioning the cutting edge just inside the top chime and then tightening the adjustment screw so that the header is held against the side of the drum. The handle of the deheader is then moved up and down while sliding the deheader along the chime. If a drum has been damaged or badly dented, it may not be possible to cut the entire top of the drum. Because it is possible that the drum is under pressure, field personnel should cut the drum a small amount to release any pressure before cutting the whole lid from the drum. Proper health and safety procedures should be followed when using a drum deheader.

10.4.3.2. Remote Opening

Two devices, discussed below, are typically used to open drums remotely, hydraulic devices and pneumatic devices.

10.4.3.2.1. Hydraulic Devices

Remotely operated hydraulic devices use hydraulic pressure to pierce the wall of the drum. These devices consist of a manually operated pump that pressurizes oil through a length of hydraulic line. The metal-pointed piercing device is attached to the end of this line and pushed into the drum by hydraulic pressure.

The piercing device attaches to the drum so that a sampling hole is made in either the side of the head or the lid. Some hollow or tube-like piercers are designed to be left in place as a permanent tap or sampling port. Piercers are designed to establish a tight seal upon penetrating the container.

10.4.3.2.2. Pneumatic Devices

Pneumatic devices use compressed air to remove drum bungs remotely. A pneumatic bung remover consists of a compressed air supply that is controlled by a heavy-duty, two-stage regulator.

A high-pressure air line delivers compressed air to a pneumatic drill, which turns the selected bung removal fitting. An adjustable bracketing system positions and aligns the pneumatic drill over the bung. The bracketing system must be attached to the drum before the drill can be operated.

10.4.4. Preparation for Cylinder Sampling

Compressed gas cylinder sampling and identification of unknown products pose great environmental and personal risks and should be performed only by persons with appropriate training and experience. A local safety authority should be contacted prior to sampling. A compressed gas supplier may also be contacted for input.

Prior to initiation of a sampling event, all cylinders should be inventoried. The following information concerning each cylinder should be recorded in the FLB/FN:

- Type of cylinder
- Contents
- Total capacity estimate
- Markings
- Labels
- Color
- Origin
- Condition.

Each cylinder should be marked with a unique identification number that corresponds to the sample container for present and future reference. Photographs of the numbered cylinders are useful for documenting the condition of the cylinder and providing a permanent record.

10.4.5. Preparation for Storage Tank, Vacuum Truck, Process Vessel, and Similar Large Container Sampling

Prior to initiation of a sampling event, all containers should be inventoried. The following information concerning each container should be recorded in the FLB/FN:

- Type of container
- Total capacity estimate
- Actual capacity (if container is open)
- Markings
- Labels
- Color
- Origin
- Condition
- Existence and condition of ladders and catwalks.

Each container should be marked with a unique identification number that corresponds to the sample container for present and future reference. Enamel spray paint is suitable for this purpose. Photographs of the numbered vessels are useful for documenting the condition of the containers and providing a permanent record.

The procedure for opening a large containment vessel will vary with different containers. Ideally, large tanks and pressure/vacuum containers should be vented and opened for sampling by the tank operator familiar with tank access processes.

Most large tanks and vacuum trucks have valves near the bottom of the tank and hatches near the top. It is preferable to collect samples from the top of a tank for several reasons. First, the integrity of valves near the bottom of the tank cannot be assured; the valve may be immobile, break, or become jammed in the open position, resulting in an uncontrolled release of the tank contents. Second, the contents of a large vessel may become stratified, and collecting a sample from the bottom will not permit the sampling of each strata. The preferred method is to access the tank from the top to obtain a cross-sectional sample of its contents.

While opening and sampling larger containment vessels, precautions should be taken to ensure personal health and safety. Accessing storage tanks requires a great deal of manual dexterity. Such sampling usually requires climbing to the top of the tank through a narrow vertical or spiral stairway while wearing protective gear and carrying sampling equipment. At least two persons should perform the sampling: one to open the hatch and collect the samples and the other to stand back (usually at the head of the stairway) and observe, ready to assist or call for help.

Prior to opening the hatch, sampling personnel should check the tank for a pressure gauge. Large tanks and pressure/vacuum containers should be opened for sampling by the tank operator familiar with tank access procedures. However, if necessary, the release valve should be opened slowly to bring the tank to atmospheric pressure. If no release valve exists, field personnel should slowly loosen hatch cover bolts to relieve the pressure in the tank. Personnel should stop immediately if the pressure is too great or if a release occurs. Hatch cover bolts should not be removed until the tank is at atmospheric pressure.

If the tank pressure is too great or venting releases gases or vapors, field personnel should discontinue venting immediately. Sampling may need to be postponed until the proper equipment is available to control the release. Any atmospheric releases should be measured with portable field instrumentation and recorded in the FLB/FN.

10.5. Sample Collection Procedures

10.5.1. Large Tanks

Prior to sample collection, ensure that tanks are stabilized, proper safety precautions are taken, and backup personnel are in place. If the contents of the tank are stratified, each stratum should be sampled discretely. At a minimum, top, middle, and bottom samples should be collected. If the container has separate compartments, each should be sampled separately at varying depths. Upon retrieval of the sample, field personnel should immediately transfer the sample into the sample container.

Depending on the objective of the sampling event (e.g., characterization for disposal), samples may be composited in the laboratory on a weight/weight or volume/volume basis prior to analysis. Knowledge of the tank-filling process will guide the decision of whether or not to composite. Hot spots and separation of materials in compartments may reduce disposal costs if only a portion of the tank is contaminated. Compositing samples may result in low reported laboratory concentrations that are not representative of overall concentration. A well-designed sampling plan will ensure sampling accuracy and provide the data necessary for decision making.

10.5.2. Secondary Containment Sampling

Secondary containment helps prevent serious environmental problems by containing storage container releases. While the storage container itself is vital to minimize the potential for leaks, secondary containment is another important safeguard. The sampling of two types of secondary containment devices, barriers and double-walled tanks, is discussed below.

10.5.2.1. Barriers

Barriers or dikes are used to contain releases from storage containers and process units. The storage containers are typically placed on an impermeable barrier such as a continuous concrete slab, lined concrete slab, or geosynthetic clay liner. For sampling inside this type of secondary containment, use one of the surficial sampling techniques discussed below.

10.5.2.2. Double-Walled Tanks and Interstitial Spaces

Double-walled tanks are another common type of secondary containment. In general, double-walled tanks do not require barriers or dykes. For USTs storing petroleum products, regulations require monitoring of interstitial spaces to determine when primary containment has failed. For USTs, liquids generally are not allowed to accumulate if systems are properly monitored. The interstitial spaces of above-ground, double-walled storage tanks often are not monitored; however, tank integrity is checked regularly. Over time, small leaks may allow liquids to accumulate in interstitial spaces. For sampling interstitial spaces, use one of the containerized liquid sampling techniques discussed in Section 10.5.5. Depending on container construction, there may be limited access to interstitial spaces for sampling.

10.5.3. Surficial Sampling

Surficial sampling is used to assess the existence or extent of contamination on surfaces rather than in a soil, liquid, or air matrix. For example, the interior of a building may be assessed by collecting wipe samples from process vessels and interiors of ventilation ducts. Though wipe, chip, sweep, and rinsate sampling have similar purposes, they are intended to assess different types of surface areas.

This recommended protocol outlines procedures and equipment for the collection of representative wipe, chip, sweep, and rinsate samples.

10.5.3.1. Wipe Samples

Wipe sampling is intended to monitor surficial contamination of non-volatile species of analytes (e.g., PCBs, metals, tetrachlorodibenzo-p-dioxin (TCDD), and tetrachlorodibenzofuran (TCDF) on non-porous surfaces, such as metal or glass. Sampling points should be chosen carefully and based on site history, manufacturing processes, personnel practices, obvious contamination, migration pathways, and available surface area. Suggested sampling points include process vessels, ventilation ducts and fans, exposed beams, and window panes. The sampling area should be large enough to provide a sufficient amount of sample for analysis (smaller sample amounts result in higher detection limits).

To collect a wipe sample, the following equipment is needed:

- Ruler or measuring tape to measure out the area being wiped
- 3-inch by 3-inch gauze pad inside a precleaned container
- Sample container
- Appropriate collection solvent
- Surgical gloves, changed prior to handling clean gauze, the sample container, or solvent.

The use of filter paper for wipe sampling is not recommended because it tends to rip and crumble on rough surfaces. If filter paper is used, it should be 4-inch-diameter heavy gauge paper, such as Whatman #4 filter paper.

Once the sample location is determined, sample collection can begin. For each sample, a new pair of surgical gloves should be worn to avoid cross contamination of the collected samples. The following procedure should be used for taking wipe samples:

1. Premeasure the area to allow for easier calculation of final results. If premeasuring is not feasible, measurements may be taken after the area is wiped.
2. Wearing a new pair of surgical gloves, remove the gauze pad from its sterile wrapping container and soak the gauze pad with the appropriate solvent.
3. Applying moderate pressure, wipe the entire sample area once in the horizontal direction and once in the vertical direction.
4. Wipe the entire sample area so that all sample material is picked up.
5. Place the gauze pad in the sample container.

A field blank should be collected for every lot of wipe material or solvent used. In some cases, blanks may be collected every 10 wipes to provide additional data for interpretation. Field blanks help identify potential contaminants from the pad, solvent, sample container, or ambient air. To perform a wipe blank, start by wearing new gloves, then wet a gauze pad with the solvent or water (for each collection parameter) and place the pad directly into the sample container.

In some cases, the laboratory should be told to rinse each sample container with an appropriate extraction or digestion solvent (depending on the analysis being performed) when transferring a sample to the extraction glassware. This procedure will ensure that the entire sample is removed from the container.

10.5.3.2. Chip Samples

Chip sampling is intended to monitor surficial contamination of non-volatile species of analytes (e.g., metals, PCBs, TCDD, and TCDF) on porous surfaces (e.g., cement, brick, and wood). Sampling points should be chosen carefully based on site history, manufacturing processes, personnel practices, obvious contamination, and available surface area. Suggested sampling points include floors near process vessels and storage tanks and loading dock areas. The sampling area should be large enough to provide a sufficient amount of sample for analysis (smaller sample amounts result in higher detection limits). To facilitate the calculations once the analytical data is received, the sample area should be measured.

To collect a chip sample, the following equipment is needed:

- Ruler or measuring tape to measure the area to be sampled
- Surgical gloves, changed prior to collecting each sample
- Decontaminated chisel of borosilicate construction and a manual or electric hammer
- Natural bristle brush and a dustpan lined with aluminum foil or one that is dedicated, decontaminated, and constructed of a pre-approved material that will not interfere with the contaminants of concern
- Sample container.

Once the sample location has been determined and marked off, sample collection can begin. The following procedure should be used for collecting chip samples:

1. Measure the sample area (for calculation of final results).
2. Break up the sample surface using a decontaminated chisel and hammer. Avoid scattering pieces beyond the sampling boundary. Any pieces that fall outside the sampling area should not be used. Chip depth should be less than ¼ inch (preferably ⅛ inch).
3. Record chip depth in the FLB/FN.
4. Collect the chipped pieces using a dedicated, decontaminated dustpan and natural bristle brush, and transfer the sample directly into the sample container.

10.5.3.3. Sweep Samples

Sweep sampling is intended to monitor surficial contamination of non-volatile species of analytes (e.g., metals, PCBs, TCDD, and TCDF) in residue found on porous (e.g., asphalt) or non-porous (e.g., metal) surfaces. Sweep sampling collects dust and residue samples that may help determine and delineate contaminants. Sampling points should be based on site history, manufacturing processes, personnel practices, obvious contamination, migration pathways, and available surface area.

Suggested sampling points include areas in ventilation systems where dust can collect, floor surfaces near process vessels and storage tanks (e.g., linoleum floors where a solvent cannot be used or too much residue exists for a wipe sample to be easily collected), and street gutters where contaminated sediments may have migrated and accumulated. The sample area should be large enough to provide a sufficient amount of sample for analysis.

To collect a sweep sample, the following equipment is needed:

- Natural bristle brush
- Decontaminated stainless steel spatula and/or a dustpan lined with aluminum foil or one that is dedicated, decontaminated, and constructed of a pre-approved material that will not interfere with the contaminants of concern
- Surgical gloves, changed prior to collecting each sample
- Sample container.

Once the sample location is determined, sample collection can begin. Wearing a new pair of surgical gloves, sweep all residues in the sample area onto the dustpan or directly into the sample container. A dedicated, decontaminated spatula may be used to aid in transferring the sample into the sample container.

10.5.3.4. Rinsate Samples

Rinsate sampling is intended to determine if surfaces contain hazardous waste residue after being cleaned. This type of surficial sampling is associated normally with drum storage pads, floors of buildings, and the interior walls of waste tanks.

Rinsate samples are performed by collecting water from the last rinse step during the cleaning of a tank or surface area. The water is analyzed for known chemical contaminants and compared against a blank sample of the same type of water.

10.5.4. Transformers

The many peculiarities associated with transformers warrant that they be considered separately from drums and tanks. Because transformers often are located in secured, out-of-the-way locations, access may be a problem. A power-operated scissor lift or cherry picker may be needed to access pole-mounted transformers. In other cases, the transformer may be in an underground cell.

Special precautions should be taken due to the toxic nature and hazards posed by PCBs, which may be present in a transformer. Appropriate protection should be worn by sampling and support personnel. To aid in spill prevention and control, make sure to use plastic sheeting and absorbent pads

WARNING: Prior to sampling, transformers must be certified as “off line” and de-energized by an electrician or other authorized person. Internal pressure should be relieved before opening the transformer.

Once power to the transformer is cut and spill control measures are in place (plastic sheeting on ground or floor surface of lift), the cover of the transformer can be removed with hand tools. A sample of the dielectric fluid can be obtained with a disposable glass composite liquid waste sampler (COLIWASA); see Section 10.5.5.2.

The following procedure should be used to obtain a representative sample from the transformer:

1. Lower the COLIWASA at a rate that allows fluid levels on both the inside and the outside of the sampler to equalize.
2. Close the COLIWASA when it reaches the bottom of the transformer and, as the COLIWASA is retrieved, wipe it with a disposable absorbent pad.
3. Transfer the sample directly into the sample container. If a disposable sampling device is used, and if the transformer is out of service, it may be possible to leave the used sampler in the sampled material; however, this should be done only after consultation with responsible authorities. Otherwise, the sampler should be drummed along with protective clothing, sheeting, and absorbent pads, and disposed of at a predetermined approved location.

Note: Never use the transformer drain valve for sample collection for the following reasons:

1. The integrity of these valves cannot be assured; the valve may be rusty, may break, or may become jammed in the open position, resulting in the uncontrolled release of transformer contents.
2. Transformer contents may be stratified. Because PCBs are heavier than other insulating oils, the resulting stratification may prevent collection of representative samples. Samples obtained from the valve near the bottom of the transformer might indicate higher PCB concentrations than the true concentration of the total dielectric fluid.

10.5.5. Containerized Liquids

Liquids stored in a container are some of the most difficult to sample. Several factors determine which method to use for sampling containerized liquids, including container location, size and location of the container opening, and type of equipment available for sampling. The health and safety of sampling personnel also plays a key role in determining sampling methodology.

No matter what type of sampler is chosen, it should allow for the collection of all phases present in the container. Containers rarely hold a homogeneous mixture of material.

Sampling devices and their procedures for use with containerized liquids are presented below. Other appropriate sampling devices include the Bacon bomb sampler, Kemmerer sampler, and weighted bottle sampler (see Chapter 6).

Sampling containerized liquids presents unique obstacles to sampling personnel. Procedures for container staging, identification, and opening should be well planned. Health and safety precautions associated with sampling containerized materials are generally more stringent. QA/QC guidelines for waste samples, as opposed to environmental samples, are unique; therefore, each site should be considered individually. If high levels of contaminants are expected, trip and field blanks may be needed. However, if residual or low-level waste or chemicals are expected in the sampled material, trip and field blanks may not be needed. If VOCs analyses are being conducted, trip and field blanks are required.

All containers should be inventoried prior to the initiation of the sampling event. The following information concerning each container should be recorded in the FLB/FN:

- Type of container
- Estimated total capacity of individual container
- Actual container capacity
- Markings, labels, color, origin, condition, etc.
- Photographs may also be taken to provide a permanent record.

Depending on the container location and position, it may be necessary to upright and/or relocate containers prior to sampling. Containers that can be moved should be positioned so that the opening, or bung, is upright, if the integrity of the container will allow. Containers should not be stacked.

WARNING: Drums containing liquid waste can be under pressure or vacuum. A bulging drum should not be moved or sampled until the pressure can be relieved safely.

The sampling of tanks, containers, and drums presents unique problems. Waste containers have various access ports including small access ports, manways, or hatches on the larger vessels or taps and bungs on smaller drums. The physical size, shape, construction material, and access location of each vessel will limit the types of equipment and methods that can be used for sample collection.

When liquids are contained in sealed vessels, gas vapor pressures build up, sludge settles out, and density layering develops. The potential for explosive reactions or the release of noxious gases when containers are opened requires considerable safeguards. The vessels should be opened with extreme caution. Certain situations may warrant preliminary sampling of headspace gasses. At a minimum, a preliminary check with an explosimeter or an organic vapor analyzer will help determine levels of personnel protection and may aid in selecting a sampling method.

In most cases, it is impossible to observe the contents of sealed or partially-sealed vessels. Since some layering or stratification is likely in any undisturbed solution, a sample should be taken that represents the entire depth of the vessel.

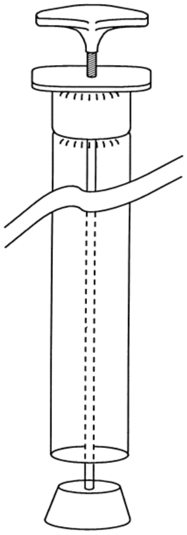
Agitation of vessel contents to disrupt the layers and rehomogenize the sample is physically difficult and almost always undesirable. In vessels greater than 1 meter in depth, the preferred sampling method involves slowly, and in known increments of length, lowering the suction line from a peristaltic pump. Discrete samples can be collected at various depths and combined or analyzed separately. If the specific depth of the sample is important, a discrete sampling device, such as the Kemmerer type, may be required. In situations where the reactive nature of the contents is known, a small submersible pump may be used.

When sampling a previously sealed vessel, a check should be made for the presence of bottom sludge by measuring the depth to the apparent bottom and then comparing it to the known interior vessel depth.

Liquid samples from containers such as 55-gallon drums may be collected using lengths of glass tubing known as drum thieves. These tubes are normally 122 centimeters in length with a 6- to 16-millimeter inside diameter. Larger diameter tubes may be used for viscous fluids if sampling with the small diameter tube is not adequate. Sampling using the drum thief requires a two-person sampling team (minimum) and proper protective gear. Use of a drum thief is simple, relatively inexpensive, quick, and does not require equipment decontamination; however, this method does have serious drawbacks. Most low-density fluids do not hold well in the glass tubes. A significant amount of the potential sample flows out of the bottom of the tube as it is raised from the drum, thereby reducing the representativeness of collected material. Many variations to this technique have been reported, including the incorporation of a small suction device (e.g., a pipette bulb) to the top of the tube, as well as the use of various tube sizes. Some success has been reported with tubes that have been heated at one end and then drawn to form a smaller orifice. This technique allows for the use of larger diameter tubing, which collects a larger sample volume per attempt, while reducing material loss from the bottom of the tube.

The COLIWASA is a sampler designed for representative sampling of multiphase wastes from drums and other containers. The COLIWASA is commercially available or can be fabricated from a variety of materials, including PVC, glass, or Teflon®. A COLIWASA usually consists of a 152-centimeter by 4-centimeter (inside diameter) tube with a neoprene stopper at one end, attached by a rod running the length of the tube to a locking mechanism at the other end. Manipulation of the locking mechanism opens and closes the sampler by lowering and raising the neoprene stopper. A recommended model of the COLIWASA is shown in Figure 10-1. However, the design can be modified or adapted to meet the needs of the sampling event.

Figure 10-1. COLIWASA Sampler



The COLIWASA is used primarily to sample containerized liquids. The PVC COLIWASA can sample most containerized liquid wastes except for those containing solvents. A glass COLIWASA is suitable for wastes that are unsuitable for the plastic unit, except for strong alkali and hydrofluoric acid solutions. Due to the unknown nature of most containerized wastes, samplers composed of glass or Teflon® are preferred in lieu of samplers containing PVC.

To identify stratified drum content and sample stratified samples, lower a glass COLIWASA into a drum all the way to the bottom slowly to allow the sample in the COLIWASA to equalize with the material in the drum, and then visually examine the withdrawn sample for stratified layers and depths. The stratified layers in the CALIWASA may be individually transferred to sample containers, or another COLIWASA may be used to collect stratified samples at the specific depths.

The major drawbacks associated with using a COLIWASA involve potential decontamination requirements and higher costs.

Specific procedures for using the drum thief and COLIWASA are provided below.

10.5.5.1. Procedures for Drum Sampling Using Glass Tubes (Drum Thieves)

The following method is recommended for using glass tubes.

1. Record the drum identification number or label for later confirmation, sampling, or disposal.
2. Open the drum.
3. Remove the cover from the sample container.
4. Lower the glass tubing until it almost touches the bottom of the container. Keep at least 30 centimeters of tubing above the top of the container.
5. Allow the sample in the tube to equalize with the material in the drum.
6. Cap the top of the tube with a safety-gloved thumb or a rubber stopper.
7. Carefully remove the capped tube from the drum, and insert the uncapped end into the sample container.

8. Release the thumb or stopper covering the tube, and fill the sample container to approximately 90% capacity or until no headspace remains for volatiles.
9. Repeat steps 2 – 7 if more volume is needed to fill the sample container.
10. Remove the tube from the sample container, and replace the tube in the drum.
11. Cap the sample container tightly with a Teflon®-lined cap, and attach the sample identification tag.
12. Replace the bung, or place plastic over the drum.
13. Place the sample container in a resealable plastic bag (one per bag). Place the bag in a clean paint can for transport.
14. Mark the sample identification number on the outside of each paint can, and complete the COC record and the FLB/FN.

The following optional method is recommended if a sample of the bottom sludge is desired:

1. Remove the covers from the container opening and the sample container.
2. Lower the glass tubing until it almost touches the bottom of the container. Keep at least 30 centimeters of tubing above the top of the container.
3. Allow the sample in the tube to equalize with the material in the drum.
4. Gently push the tube toward the bottom of the drum into the sludge layer. Do not force it.
5. Cap the top of the tube with a safety-gloved thumb or rubber stopper.
6. Carefully remove the capped tube from the drum, and insert the uncapped end into the sample container.
7. Release the thumb or stopper covering the tube, and allow the sample container to fill to approximately 90% capacity or until no headspace remains for volatiles. If necessary, the sludge plug in the bottom of the tube can be dislodged with the aid of a stainless steel laboratory spatula.
8. Repeat steps 2 – 7 if more volume is needed to fill the sample container, and recap the tube.
9. Proceed as in steps 10 – 13 above.

Note: If a reaction (e.g., violent agitation, smoke, and/or light) is observed when the glass tube is inserted, field sampling personnel should evacuate the area immediately. If the glass tube becomes cloudy or smoky after insertion into the drum, then hydrofluoric acid might be present. Therefore, a comparable length of rigid plastic tubing should be used to collect the sample. When a solid is encountered in a drum (either in a layer or as bottom sludge), the method described above for sampling bottom sludge may be used to collect a core of the material. The material may also be collected with a disposable scoop attached to a wooden or plastic rod.

10.5.5.2. COLIWASA Use Procedures

The following procedures can be adapted for use with other versions of COLIWASA samplers:

1. Select the COLIWASA material, assemble the sampler, and ensure the sampler is clean.
2. Check to make sure the sampler is functioning properly. If necessary, adjust the locking mechanism so the neoprene rubber stopper provides a tight closure.
3. Wear the necessary PPE, and observe required sampling precautions.
4. Open the sampler by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.
5. Slowly lower the sampler into the liquid waste at a rate that permits the levels of the liquid inside and outside of the sampler tube to equalize. If the liquid level in the sample tube is lower than the liquid level outside the sampler, the sampling rate is too fast and will result in a non-representative sample.
6. 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 posi-

tion by turning the T-handle upright and resting one end tightly on the locking block.

7. Slowly withdraw the sample from the waste container with one hand while wiping the sampler tube with a disposable cloth or rag with the other hand.
8. Carefully discharge the sample into a suitable sample container by slowly pulling the lower end of the T-handle away from the locking block with the lower end of the sampler positioned in the sample container.
9. Cap the sample container with a Teflon®-lined cap, label the container, and complete the COC record and the FLB/FN.
10. Unscrew the T-handle of the sampler, and disengage the locking block. Clean the sampler on site, or store the contaminated parts of the sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

10.5.6. Waste Pile Sampling

The protocol discussed below outlines general procedures for collecting samples from waste piles and other waste materials, equipment necessary for sampling, and adequate representation of the material. This section also discusses factors to consider when formulating a sampling plan. Due to the many variables associated with waste material sampling, exact procedures cannot be outlined for every sampling situation. These variables include the following: shape and size of piles; size, compactness, and structure of the waste material; and make-up. To determine the correct sampling methodology, the chemical and physical properties of a sample, the purpose of the sampling event, analysis procedures, and the intended use for the analyzed data should all be considered when planning the sampling event.

10.5.6.1. Considerations for the Sampling Plan

The physical and chemical make-up of the waste pile and the purpose for sampling should be considered when planning the sampling event. Considerations for creating a waste pile sampling plan are presented below.

10.5.6.2. Shape and Size

The shape and size of waste material and waste piles can vary greatly. Piles may be cone-shaped, long and rectangular, square, oval, or irregularly shaped. State and federal regulations often require a specific number of samples per volume of waste; therefore, size and shape may be used to calculate volume and plan for the correct number of samples. Shape also should be considered when planning physical access to the sampling point and selecting the type of equipment necessary to safely and successfully collect the sample at that location.

10.5.6.3. Sampling Procedures

Waste pile samples can be collected at the surface or at depth. These samples generally are collected using one of the following samplers: trowel or scoop, waste pile sampler, sampling trier, soil auger, grain sampler, split-spoon sampler, or soil coring device. Surface samples can be collected most efficiently with a trowel or a scoop. For samples at depth, augers may be used. For a sample core, waste pile samplers or grain samplers may be used.

For surface sampling, clear surface debris and use a trowel or scoop to collect a sample at a depth of 0 – 6 inches. The depth of surface samples may be determined by federal, state, or local regulations. Typically, the top 6-inch soils should not be analyzed for VOCs due to evaporation and loss. For surface core sampling, use a waste pile sampler, trier, or similar sampling device. Transfer the sample directly into the sample container with a decontaminated trowel or spatula (if necessary). A wide mouth bottle is preferred because it requires less disturbance of the sample when it is transferred into the sample container. If homogenization will be performed in the field, it should be performed prior to transferring the material to sample containers.

When sampling at depth, clear away the initial top layer of material prior to taking samples, and advance the hole to the desired sampling depth with a decontaminated bucket auger or power auger. Use a separate decontaminated sampler to collect the sample, and, if necessary, a decontaminated spatula to transfer the sample into the sample container.

Sampling devices most commonly used for small waste piles include thieves, triers, and shovels. Evacuation equipment, such as backhoes, can be useful for sampling larger waste piles.

A thief consists of two slotted concentric tubes, usually made of stainless steel or brass (see Figure 10-2). The outer tube has a conical pointed tip that permits the sampler to penetrate the waste pile. The inner tube is rotated to open and close the sampler. A thief is used to sample dry granules or powdered wastes, where particle diameter is less than one-third the width of the slots.

Figure 10-2. Thief Sampler

The following procedure is used to collect samples using a thief sampler:

1. While the sampler is in the closed position, insert it into the sample material from a point near a top edge or corner, through the center, and to a point diagonally opposite to the point of entry.
2. Rotate the inner tube of the sampler into the open position.
3. Wiggle the sampler a few times to allow materials to enter the open slots.
4. Place the sampler in the closed position, and withdraw it from the material being sampled.
5. Place the sampler in a horizontal position with the slots facing upward.
6. Rotate and slide the outer tube from the inner tube.
7. Transfer the sample into a laboratory-cleaned sample container, label the container, and complete the COC record and the FLB/FN.
8. Clean the sampler on site, or store the contaminated parts of the sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

A trier consists of a tube cut in half lengthwise with a sharpened tip that allows the sampler to cut into sticky solids and to loosen soil (see Figure 10-3). A trier samples moist or sticky solids with a particle diameter less than one-half of its diameter. Triers are available from 61 – 100 centimeters in length and from 1.27 – 2.54 centimeters in diameter. Larger triers can be fabricated.

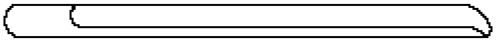
Figure 10-3. Sampling Trier

The following procedure is used to collect samples using a sampling trier:

1. Insert the trier into the material to be sampled at a 0- to 45-degree angle from horizontal. This orientation minimizes spillage of sample.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. Transfer the sample into a laboratory-cleaned sample container using a spatula. Record applicable data in the FLB/FN, and complete the sample analysis request sheet and COC record.
5. Clean the sampler on site, or store the contaminated parts of the sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

Waste pile samplers are used for sampling wastes in larger heaps with cross-sectional diameters of greater than 1 meter (see Figure 10-4). They also can be used for sampling granular or powdered wastes, materials in large bins or barges, or soils where the grain sampler or sampling trier is not long enough. The waste pile sampler essentially is a large sampling trier. It is commercially available or easily fabricated from sheet metal or plastic pipe. Procedures for using the waste pile sampler essentially are identical to the sampling trier.

Figure 10-4. Waste Pile Sampler



10.5.6.4. Characteristics of the Material

Materials to be sampled may be homogeneous or heterogeneous. Homogeneous material, such as sand blast abrasive, may not require an extensive sampling protocol. The DQOs of a sampling event will determine whether grab or composite sampling is required. If a positive identification is made in analyzing a composite sample, then the discrete samples should be analyzed individually to determine the true distribution of contaminant throughout each component of the composite. A thief or trier sampler (if practical for the particular application) would provide a more representative sample compared with a shovel.

If the waste pile is composed of heterogeneous material, try to determine the source, nature, and extent of the deposited waste. If possible, sample the waste sources contributing to the pile to determine the characteristics of the waste pile. If any of the waste within a heterogeneous waste pile is hazardous, the entire waste pile should be managed as hazardous waste, unless the waste pile can be separated into hazardous and non-hazardous components.

Waste materials may be affected by their inherent chemical stability. Weathering and leaching over time may cause older material to differ in chemical composition from newly deposited material in the same pile. Heterogeneous material may undergo chemical reactions resulting in pockets or layers of different compounds.

Waste piles containing unknown waste can be sampled using a three-dimensional random sampling strategy. This strategy involves dividing the pile into a three-dimensional grid system and numbering each grid section and sampling point using random-number tables or random-number generators. This strategy should be used only if all points within the pile can be accessed.

10.5.6.5. Composite Samples

Compositing a number of individually collected field samples may help reduce analytical costs. The disadvantage of sample compositing is a loss of concentration variance data, whereas the advantage is a more representative sample from the population that is being sampled. Compositing is especially useful if the waste pile is homogeneous. Compositing requires that equal amounts of material from each sample be combined, and ground, sieved, and/or sufficiently mixed so that an aliquot sample can be removed for laboratory analysis. To improve the quality of the composite sample, follow the compositing considerations offered in ASTM D6051, *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities*.

If the concentration of a regulated material exceeds regulated levels, the entire waste pile should be managed as regulated waste, or additional sampling and analyses should be performed to define regulated and non-regulated material.

To avoid having to resample, field sampling personnel should collect enough volume to provide for a different compositing scheme or additional individual analyses. This precaution requires initial field samples to be retained until the laboratory results on the composited sample become available.

When a waste pile is believed to be heterogeneous, individual random sampling is the preferred sampling technique.

10.5.7. Containerized Solids

Sampling containerized solid materials (e.g., sludge, granulars, and powders) is accomplished generally with one of the following samplers:

- Scoop or trowel
- Waste pile sampler
- Grain sampler.

Each sample should be taken discretely. Depending on the objective of the sampling event (e.g., characterization for disposal), compositing samples in the field or laboratory on a weight/weight basis prior to analysis may be permissible. The procedure to collect samples using a waste pile sampler is discussed in the previous section.

Use the following procedure to collect samples using a scoop or trowel:

1. Open the container to be sampled.

2. At specified intervals, take small, equal portions of sample from the surface and immediately below the surface.
3. Transfer the sample into a laboratory-cleaned sample container, label the container, and complete the COC record and the FLB/FN.
4. Clean the sampler on site, or store the contaminated sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

A grain sampler consists of two slotted telescoping tubes, usually made of brass, stainless steel, or high-density polyethylene. The outer tube has a conical, pointed tip on one end that permits the sampler to penetrate the material being sampled. The sampler is opened and closed by rotating the inner tube. Grain samplers are generally 61 – 100 centimeters long by 1.27 – 2.54 centimeters in diameter. The following procedure is used to collect samples using a grain sampler:

1. Open the container to be sampled.
2. While the sampler is in the closed position, insert it into granular or powdered material or waste being sampled from a point near a top edge or corner, through the center, and to a point diagonally opposite the point of entry.
3. Rotate the inner tube of the sampler into the open position.
4. Wiggle the sampler a few times to allow materials to enter the open slots.
5. Place the sampler in the closed position and withdraw from the material being sampled.
6. Place the sampler in a horizontal position with the slots facing upward.
7. Rotate and slide out the outer tube from the inner tube.
8. Transfer the sample into a laboratory-cleaned sample container, label the container, and complete the COC record and the FLB/FN.
9. Clean the sampler on site, or store the contaminated sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

10.5.8. Sampling for Lead in Paint

X-ray fluorescence spectrometry and direct laboratory testing may be used to determine the lead content of paint. However, X-ray fluorescence spectrometry is generally used only as a screening test to detect the presence of lead, because certain interferences can make it unreliable for determining exact concentration. Laboratory testing should be used to determine the actual concentration of lead in solid materials.

To collect a paint sample suitable for lead determination, use a sharp knife to cut through the paint. Remove the paint with a clean putty knife, and place the sample in a clean resealable plastic bag or appropriate container. Label the plastic bag or container and record pertinent sample collection information in the FLB/FN.

A sample of about 2 inches by 2 inches is required for a determination of lead content. Be sure to take a sample of all layers of paint, because only the lower layer may contain lead. Avoid including any of the underlying wood, plaster, metal, or brick in the sample. Wipe the sampled surface and any paint dust with a wet cloth or paper towel, and discard the cloth or towel.

If it is determined that a painted surface is homogeneous throughout the building and the same paint was applied at the same general time throughout, the number of samples can be reduced. At a minimum, three or five samples each from doors, windows, and walls for each floor of a building should be collected.

The U.S. Department of Housing and Urban Development recommends that action be taken to reduce exposure to lead in paint when the lead content is greater than 0.5% by laboratory testing or greater than 1.0 mg/cm^2 by X-ray fluorescence spectrometry. When the lead content of a door, window, or wall sample exceeds the recommended action level, all similar areas should be inspected. Action is especially important when paint is deteriorating or when infants, children, or pregnant women are present. Generally, buildings constructed after 1980 are not expected to contain paint with elevated lead concentrations.

Lead-contaminated waste generated by public, commercial, and industrial buildings is subject to hazardous waste regulations. Analysis of the waste using the Toxic Characteristic Leaching Procedure (TCLP) may be required. Lead-contaminated wastes with TCLP levels at or above 5 ppm are defined as hazardous and should be handled and disposed of in conformance with hazardous waste laws and regulations.

When lead-based paint is an integral part of demolition debris, the appropriate way to collect samples is by coring a representative portion of the material to be disposed to include both paint and wood. Because a relatively small amount of lead is present in the paint film, these wastes may not fail TCLP, and many can be disposed of as solid waste. If the waste is lead-based paint chips or blasting wastes, TCLP should be used on the waste to determine if it is hazardous. Due to the more concentrated nature of these wastes, they will often fail TCLP and require management as a hazardous waste.

10.5.9. Ashore Oil Spill Sampling

This section discusses methods for collecting surface ashore samples whenever an oil spill has occurred from a site or a vessel. A suction device can be used to collect samples. However, the statistical design of the sampling program should be the same no matter what devices are used to collect samples. Ashore oil spill samples can be collected by inverting a 4-fluid-ounce jar and collecting the liquid oil sample and store the sample at ≤6°C. Tarballs should be collected in an appropriately sized sample container and stored at ≤6°C. Shallow pollution samples can be collected by using one of the following methods.

10.5.9.1. Soil Punch Sampling

A soil punch is a thin-walled tube, 15 – 20 centimeters long, used to extract short cores from contaminated locations at beaches. The following steps are used to collect samples with a soil punch:

1. Drive the tube into the potentially contaminated location with a wooden mallet.
2. Extract the tube and core.
3. Push the collected materials into a mixing bowl, place the sample in a labeled container with a Teflon®-lined cap, and store the sample at 0 – 6°C.
4. Complete the COC record and the FLB/FN.
5. Clean the sampler on site, or store the contaminated parts of the sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

10.5.9.2. Scoop or Shovel Sampling

Surface samples following an ashore oil spill are collected with a scoop and shovel. A small plastic disposable scoop can be used to collect representative samples. Proper QA/QC procedures should be followed to ensure cross-contamination cannot occur during sampling events. Larger representative samples can be collected using a shovel.

10.5.9.3. Hand Auger Sampling

Shallow surface ashore oil spill samples can be collected with a hand auger. Samples should not be used for the examination of a core sample, because soil is mixed when the auger is pushed into the ground. The following procedure is used to collect a sample with a hand auger:

1. Place the hand auger at the location where the sample is to be collected.
2. Twist or screw the auger, extract the sample, and pull the auger.
3. Push collected materials into a mixing bowl, place the sample into a labeled container with a Teflon®-lined cap, and store the sample at ≤6°C.
4. Complete the COC record and the FLB/FN.
5. Clean the sampler on site or store the contaminated parts of the sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

Note: Power augers can be used to collect samples. The procedure is the same as for the hand auger.

10.6. QA/QC

See Chapter 3 of this handbook for information on establishing an appropriate QA/QC protocol.

A QA/QC plan involves the establishment of a sampling protocol, which is designed to minimize sources of error in each stage of the process, from sample collection to analysis and reporting of analytical data. See Chapter 2 for information on creating a SAP. Key elements of a SAP for waste sampling are as follows:

- Development of a statistically sound sampling plan for spatial and temporal characterization of the drums, waste pile, and tanks
- Installation of a vertical and horizontal sampling network to allow for the collection of subsurface samples
- Use of a sampling device that minimizes disturbance to the chemistry of the media formation
- Use of field decontamination procedures to minimize cross-contamination during sampling
- Collection of QA/QC samples and adherence to COC procedures.

10.7. Sampling Equipment List

Section 3.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional equipment applicable to waste sampling. In most cases, only a portion of the equipment listed below would be required for a given sampling event.

- Manual Drum Opening
    - Bung wrench
    - Drum deheader
- Remote Opening
    - Hydraulic device
    - Pneumatic device
- Ashore Oil Spill Sampling
    - 4-ounce jar
- Shallow pollution sampling
    - Soil punch
    - Scoop or shovel
    - Hand augers or power auger
- Wipe Sampling
    - Ruler or measuring tape
    - Gauze pad inside a pre-cleaned container and/or 4-inch diameter heavy gauge paper, such as Whatman #4 filter paper
- Chip Sampling
    - Ruler or measuring tape
    - Decontaminated chisel of borosilicate construction
    - Manual or electric hammer
    - Natural bristle brush
    - Dust pan lined with aluminum foil or one that is dedicated, decontaminated, and constructed of a pre-approved material that will not interfere with the contaminants of concern
    - Aluminum foil
- Sweep Sampling
    - Natural bristle brush
    - Decontaminated stainless steel spatula and/or a dust pan lined with aluminum foil, or one that is dedicated, decontaminated, and constructed of a pre-approved material that will not interfere with the contaminants of concern
    - Aluminum foil
- Transformer Sampling
    - Disposable glass COLIWASA
- Containerized Liquids/Drum Sampling
    - Bacon bomb, Kemmerer sampler, weighted bottle sampler, drum thief (glass tubes), and/or COLIWASA
    - Disposable scoop attached to a length of wooden or plastic rod
- Waste Pile Sampling
    - Thieves, triers, or shovels
- Containerized Solids Sampling
    - Scoop or trowel, waste pile sampler, or grain sampler
- Sampling for Lead in Paint
    - X-ray fluorescence spectrometer
    - Sharp knife to cut through the paint
    - Clean putty knife
    - Resealable plastic bag or appropriate container.

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Chapter 11. Air Sampling

11.1. Purpose

This chapter provides supporting background information and general procedures for air sampling related to environmental restoration and compliance issues.

11.2. Scope

Because the requirements and methods for air sampling are often very complex, this information is intended only to provide an overview of methods, equipment, and instrumentation. For specific applications, the established regulatory or literature reference procedures should be consulted.

11.3. Background

Air sampling is conducted to monitor levels of air contaminants. Air is the most transient environmental medium and subject to extreme spatial and temporal heterogeneity. Representative air sampling presents unique and difficult challenges. Also, the contaminants of interest may be present at very low concentrations.

11.3.1. Air Matrices

Air sampling matrices include:

- Ambient (outdoor) air
- Indoor air
- Point sources (stacks, exhausts, and other emission sources)
- Fugitive emissions (sources of air pollutants other than stacks or vents)
- Soil gas, particularly over and around landfill waste sites, or groundwater plumes (soil gas sampling is addressed in Chapter 4, Soil Sampling).

In general, ambient or outside air will contain the smallest concentration of detectable air pollutants, and indoor air will contain an increased concentration range of pollutants. Pollutant concentrations from point sources such as stacks and vents, and fugitive emissions can vary from ppb levels to almost 100% pollutant.

11.3.2. Objectives of Air Sampling and Monitoring

Air sampling and monitoring may be performed to**:**

- Determine background levels of contaminants
- Provide continuous monitoring of atmospheric or industrial process conditions
- Measure acute or periodic releases of contaminants
- Provide data for computerized dispersion modeling to evaluate contaminant patterns and trends
- Vapor intrusion (VI) investigations. See the *DoD Vapor Intrusion Handbook* for more information on conducting VI investigations
- Monitor and evaluate remediation processes (e.g., air stripping, soil vapor extraction, and excavation). See US Army Corps of Engineer’s EP-200-1-24, *Air Pathway Analysis for the Design of Remedial Action Projects*, and Chapter 11 of this Handbook and for more information.

The basic objective of air monitoring is to representatively measure in situ, or transfer all of the contaminant from a measured quantity of air into an analytical instrument that will respond proportionally to the amount of contaminant present. In practice this includes:

- Taking a portable instrument to the contaminated air location
- Taking a measured volume of the contaminated air to an instrument in the laboratory or in the field
- Extracting the contaminant from a measured volume of air and measuring its concentration in the laboratory or in the field.

Accurate measurements are often difficult to achieve, because many factors affect air sample representativeness, quantitation, and minimization of artifacts. Artifacts are contaminants originating from sources other than the intended sample. Common sources of artifacts are:

- Contaminated sampling equipment
- Improper sampling procedures

- Undesired physical or chemical reactions of the sample with the sampling container or collection media
- Thermal effects
- Contamination from adjacent sites or sources not representative of the site (e.g., indoor sources during a vapor intrusion investigation)
- Emissions from surrounding activities.

In addition, artifacts and poor quantitation are common problems with air analyses because the concentrations of contaminants are often very low with respect to the surface areas of the containers and sampling media used for their collection. Many different methods have been developed for measuring air contamination. The choice of method for a particular task depends on the anticipated use of the data, required detection/quantitation limits, data turnaround needs, data quality objectives, and regulatory requirements written into facility operating permits.

11.4. General Classification of Air Pollutants

Generally, air pollutants are broadly classified into two types – gases/vapors and particulates. Gases such as sulfur dioxide, nitrogen oxides, and carbon monoxide result primarily from industrial combustion processes. Hydrogen sulfide and halogens and their derivatives come mainly from industrial operations. Solvent vapors have numerous anthropogenic sources and can be present from primary or secondary sources (vadose zone and/or ground water contamination). Hydrocarbons and their oxidation products are primarily associated with gasoline fuel operations and incomplete combustion. Ozone results from photochemical reactions with nitrogen oxides, hydrocarbons, and other organic compounds. Particulates can be either droplets of liquid or solid particles. When particulate matter is suspended in air, it is called an aerosol. Aerosols most prevalent in pollution are in the 0.01 – 100 micron diameter particle size range. Aerosols can be formed by:

- Water vapor (e.g., fog or steam)
- Smoke and industrial fumes
- Reaction of species such as sulfur dioxide with water
- Growth of smaller entities such as salts into agglomerates (e.g., ammonium sulfate and nitrate)
- Organic liquid particulates from the distillation or vaporization of volatile materials
- Atomization of liquids through a small orifice.

Types of solid particulates include:

- Soot or carbon found in smoke from combustion processes of natural or man-made origins (particles are 0.01 – 1.0 micron diameter and very sticky)
- Fly ash consisting of non-combustible mineral solids formed from complete oxidation of coal or fuel oil, generally <200 micron diameter
- Dusts from natural or industrial disintegration processes, generally 1 – 1,000 micron diameter
- Fumes from condensation, calcination, or chemical reactions.

Liquid particulates include:

- Mists arising from vapor condensation, chemical reactions, or liquid atomization.

Many common types of pollutants are actually combinations of the general classifications:

  - Smoke is a combination of gases and solid and liquid particulates
  - Solid particulates frequently have organic and inorganic species adhered to their surfaces
  - Liquid particulates may combine with gases and solid particulates
  - Atmospheric aerosols are frequently mixtures of soluble and insoluble materials, with relative humidity determining the equilibrium of the two phases. They may be chemically complex with volatiles and several soluble aerosols co-dissolved. Figure 11-1 illustrates relative particle size ranges for aerosols, dusts, and fumes.

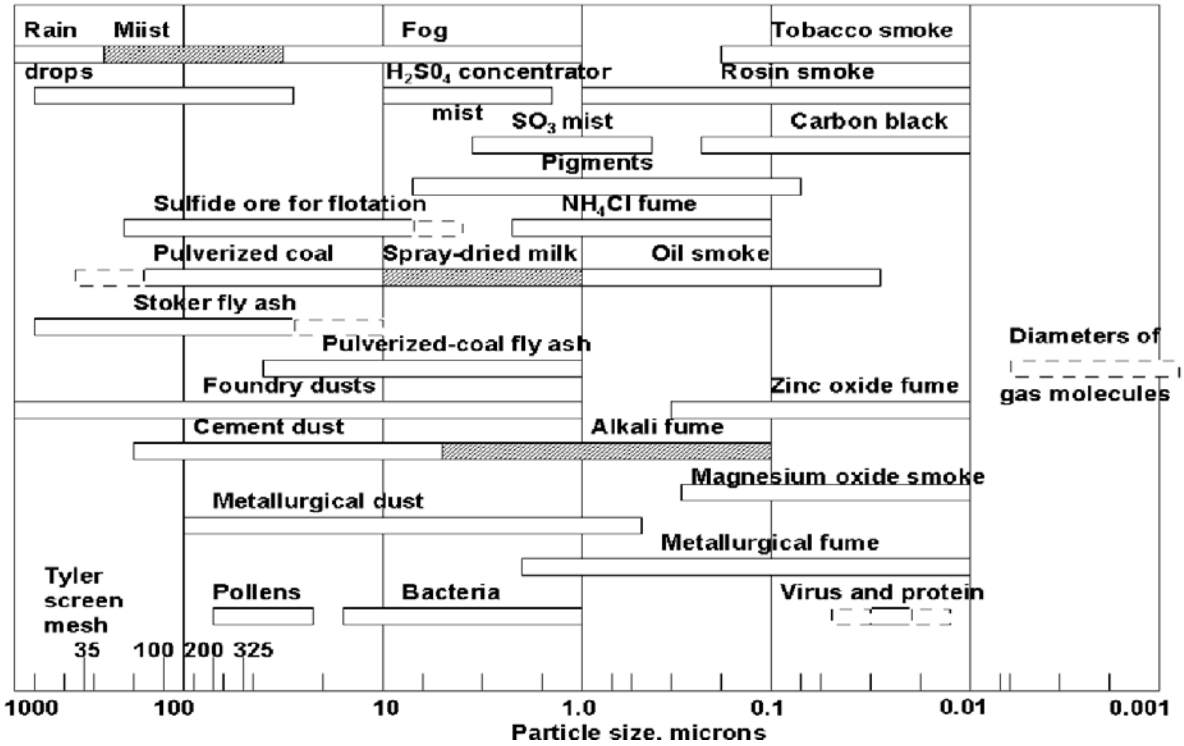

Figure 11-1 Particle Size Ranges for Aerosols, Dusts, and Fumes
Source: Faith, W. L. and Atkinson, A. A., Jr

11.5. Factors Affecting Ambient Air Sampling and Monitoring

11.5.1. Meteorological

11.5.1.1. Meteorological Parameters

Primary meteorological parameters to be considered in ambient air sampling are:

- Wind direction
- Wind speed
- Temperature
- Atmospheric stability
- Atmospheric pressure
- Precipitation.

Wind direction is most critical and must be monitored and documented constantly during air sampling events. Large variations in samples can result from even momentary shifts in wind direction. Changes in wind speed alter the volatilization rates of liquid contaminants and concentrations of contaminants downwind of the source. Generally, increased wind speed increases volatilization of liquids at the source and may conversely decrease the concentration of volatile downwind of the source. Increased wind speed may have the opposite effect on non-volatiles by increasing the amounts adsorbed by particulates such as soil and dust. Increased wind speed facilitates transfer of a greater number of large particulates from the source.

Higher temperature also increases volatilization. Both air temperature and solar radiation are factors, with solar radiation usually most critical, particularly for compounds with high vapor pressures.

Atmospheric stability refers to vertical motions of the air. When atmospheric conditions are unstable, pollutants become more dispersed in the various vertical layers. Under stable conditions, downwind concentrations are generally higher. When monitoring sources such as landfills, atmospheric pressure is a significant factor, affecting the migration of gases through and out of the source. Low atmospheric pressures favor increased volatilization rates. Precipitation decreases overall airborne contaminants. The highest concentrations of contaminants generally occur in the initial precipitation samples. This is a result of particulate matter with its precipitate and gaseous contaminants being physically scrubbed from the air by the precipitation. Precipitation resulting in 100% relative humidity also significantly decreases particle transport by wind and volatilization rates.

11.5.1.2. Meteorological Measurements

For many air dispersion modeling applications, meteorological parameters can be obtained from the National Weather Service, the National Oceanic and Atmospheric Administration (NOAA), National Climatic Data Center, and the Federal Aviation Administration (FAA). These may be hourly observations, or may be readings taken every 5 – 15 minutes and calculated to an hourly average. For certain applications, it may be necessary to set up a custom on-site monitoring program. This is important if real-time analysis is needed or if wind flow is significantly affected by the terrain.

11.5.1.3. Wind Measurements

Wind direction and speed are critical parameters in predicting contaminant path of travel, air concentration, and deposition rates. Frequently, wind measurements are made at a height of 10 meters above ground or equal to the height of the stack or source being measured. It is very important that the structure supporting the monitoring equipment does not in any way interfere with the wind measurement, nor should any other structure or topographical factors be close enough to interfere with accurate measurements. The sensors are generally mounted on a thin mast above the tower at a height three times the lateral tower dimension.

Either prop vane or rotating cup anemometers can be used to measure wind speed. The cup anemometer has the advantage of being independent of direction, and can be calibrated to within 1% accuracy. Slowing of wind speed with this type sensor can lead to 5 – 10% error if the sensor continues to rotate. Vane type sensors must be damped to prevent over rotation, which results in an artificially high directional variation.

11.5.1.4. Temperature and Humidity

For the purposes of most modeling, temperature and humidity measurements that are representative of hourly periods can be used. The sensors can be located at the height of the shelter or about 1.5 meters. For plumes, temperature is used to calculate the plume rise from buoyant sources. If the contaminants of interest are also hygroscopic, a humidity measurement is also required. Sensors should be protected from direct solar radiation and the buildup of heated air within the shielded area. This can often be done by aspirating the air flow over the sensors.

Sometimes it is desirable to measure the temperature at two levels, usually between 2 and 10 meters, and use the temperature difference to determine the degree of atmospheric stability or surface inversion.

For dry deposition measurements, it is often necessary to know air humidity, surface wetness, amount and type of precipitation, and the chemistry of the precipitation.

11.5.2. Topographical Factors

Mountains, hills, valleys, lakes, and seas can significantly affect wind direction and the dispersion patterns of air pollutants. Wind generally flows along the axis of valleys with bi-directional distribution. At night, colder air flows down the slope into the valley creating thermal inversion of the air. The result is increased pollutant concentrations in the valley due to decreased vertical air dispersion. During the daytime hours, the effect is reversed but to a lesser degree.

Near oceans and large lakes, the differences in daytime and nighttime temperatures result in bi-directional wind changes. During the day, the wind generally moves from the cooler body of water toward the land in an upward motion. At night as the land mass cools, the effect is reversed producing "land breezes", which are not as strong.

11.5.3. Extraneous Contaminant Sources

Extraneous background contamination can come from many sources. Incidental atmospheric contaminants such as automobile exhaust or exhaust from a gasoline powered generator can render samples useless. Incidental contaminants on the large surfaces of containers and sorbents used to collect or concentrate samples can also pose significant problems. Because inadvertent contamination can affect analytical results, it is imperative that all sampling equipment be scrupulously clean and many types of blanks run.

11.5.4. Response Time of Sensors

Response time is the time required for a real-time sensor, recording in situ measurements, to respond to an incremental change in contaminant concentration and settle to a final value within a margin of error of the correct contaminant concentration. This is an important criterion to consider when selecting monitoring equipment for a specific application.

11.5.5. Placement of Samplers and Monitors

Placement requirements for sampling devices and monitors vary greatly depending on the type of source, type of monitoring, data quality objectives, and duration of the sampling or monitoring event. In general, care should be taken to maximize the representativeness of the data and minimize artifacts and effects resulting from poor collector or sensor placement.

When collecting air samples to determine the effect of an operation or facility on air quality, there must be at least one upwind sample taken to determine background ambient air conditions. The arrangement of downwind samples should vary depending on site conditions and project objectives.

Since the movement of airborne contaminants depends on weather conditions, the weather must be monitored during sampling. If there is a significant change in wind direction, samplers must be moved to maintain the proper upwind-downwind arrangement.

Because sampling equipment is often exposed to the elements, and operates for extended periods of time, it should be checked frequently to ensure proper operation. It should also be placed in a secured area when possible.

11.6. General Terms

11.6.1. Whole Air versus Component Samplers

Whole air samples are collected in various types of plastic bags, glass globes, or metal canisters with polished inside surfaces. Component samples are collected by passing the air stream through a filter device, a trapping liquid, or a solid sorbent medium.

11.6.2. Real Time versus Integrated or Composite Samplers

Field or "real-time" sampling refers to a variety of methods, using specific detectors, which can quickly survey an area of interest. Often these measurements are used to determine the placement of time integrated samplers, or other samplers for analysis in a laboratory. A grab sample may also be referred to as a real-time sample since the duration of collection is usually under a minute.

Composite or time-integrated samplers collect a sample or data over a specified time period, either continuously or periodically and give a representation of air contaminants over a much longer period of time than a real-time or grab sample.

11.6.3. Active Versus Passive Samplers

Samples may be collected in either an active or passive mode. Active air samplers use a pump or vacuum to pull ambient air through the monitor or collection media. Passive samplers allow ambient conditions to diffuse the sample through the monitor or the solid adsorbent material, which may be covered with a membrane or plastic film. The samples diffuse through the membrane and are adsorbed onto the solid media.

11.7. Strategies for Assessment of Air Pollution Emissions

Several approaches exist for evaluating emission of air pollutants. The method of choice depends on the end use of the data, data quality objectives, availability of sampling equipment, accessibility of sources, and recommendations of the regulatory agencies. EPA has published a four volume series for air pathway analysis (APA). These documents, *Air/Superfund National Technical Guidance Study Series, Vols. 1 – 4* are recommended for details on the various sampling methods. *Sampling and Analysis of Airborne Pollutants*, edited by Eric D. Winegar and Lawrence H. Keith, also provides guidance in this area.

11.7.1. Direct Assessment

Generally, it is preferable to measure emission rates directly rather than determine downwind mass/volume concentrations, the reason being that the data has more versatility for use in various modeling applications, where changes in parameters such as meteorological conditions can be evaluated. Direct measurements are also more useful for determining long-term effects, determining National Priority List (NPL) status for pollutants, and meeting permitting requirements. They are most useful for volatile contaminants. The preferred EPA method for these measurements is to set up an isolation flux chamber or enclosure over a small surface area of the emission site. This method is recommended for:

- Land surfaces
- Non-aerated, non-mixed liquid surfaces
- Aerated, mixed liquid surfaces
- Fugitive emissions from processes including leaks in seams, valves, ports, and controllers.

Advantages include:

- Cost effective
- Simple equipment requirements
- Direct measurements
- No models or estimates required
- Generally better quality data
- No upwind contamination and fewer meteorological interferences.

Disadvantages include:

- Not always possible
- Primarily used for volatiles materials
- Must exercise caution in data extrapolation.

11.7.2. Indirect Assessment

The indirect assessment approach uses a variety of samples from different ambient locations relatively close to the source to estimate concentrations via dispersion modeling equations. There are many models available, some developed for stack emissions, and others for area sources. The type of model determines the placement of samplers and weighting of sample data. The models are designed to determine parameters such as downwind concentrations (sometimes at different heights), concentrations directly over the source, and general area concentrations.

Advantages include:
- Can be done where direct measurements are not feasible or possible
- Virtually any volatile or particulate species can be evaluated
- Not dependent on emission measurements
- Analyte measurements depend on analysis technique and sampling media.

Disadvantages include:
- Meteorological conditions play a major role and must be relatively stable for valid data collection
- Possibility of upwind contamination
- Lower concentrations of analytes due to mixing and dispersion forces
- Possibility of air flow interferences by topographical features
- Can be costly to perform
- Complexity of sampling devices
- Long term sampling may be difficult due to changing meteorological and atmospheric conditions
- Labor and analysis intensive
- Much more difficult to evaluate specific areas within a site.

11.7.3. Air Monitoring and Dispersion Modeling

In this method, measurements are generally made further downwind of the source and the data are used in a customized site-specific model to estimate overall site concentrations. For this type of determination to be accurate, meteorological and topographical factors must be accurately evaluated in the model. Modeling guidance can be found in the *EPA Superfund Exposure Assessment Manual, Appendix J*, which gives a brief description of EPA and other air pollution models. After a model is chosen, an estimate is made of source emission and downwind concentrations. The predicted and actual downwind concentration data are compared and source concentration is iteratively varied within the modeling equations until the data fit the equations for source and downwind concentrations. From the iterative process, the actual source emission concentration is thus estimated.

Advantages include:
- Can be done at greater distance from the source
- Exact data on source emission not required.

Disadvantages include:
- Dispersive forces result in concentrations being lower further from source
- The fit of the model to the actual conditions is critical
- Because the process is iterative, the uncertainty may be large.

11.7.4. Predictive Modeling

This method is based on choosing an emission model for a specific source type. Data for these models are usually obtained from literature, estimated, or calculated. For better accuracy, some data may need to be determined experimentally. The method is useful when used for screening or when an assessment does not require a high degree of confidence.

Advantages include:
- Fast
- Inexpensive
- Requires fewer or no field measurement
- Useful screening tool.

Disadvantages include:
- Provides estimate only
- Fit of site source and model critical
- Can be very inaccurate.

11.8. Field Measurement Instruments

Instruments have been developed for field operations to allow real-time measurement of air quality. Most instruments are meters, which measure analyte concentrations by passing a stream of air through the detector for a finite time interval. Other instruments monitor continuously and sound an alarm when a pre-set concentration level is exceeded, or monitor over a given time period and determine a time weighted average exposure. Before using any field instruments, the following factors must be considered:

- Limitations based on functional design or manufacturer quality control
- Safety must always be considered to ensure that the chosen instrument is compatible with the potential hazard. For example, some instruments are capable of detecting explosive hazards, but not all are safe for operations under these conditions. If the atmospheric concentration is potentially greater than 25% of the lower explosive limit, the meter itself must be certified safe for operation. Agencies certifying equipment safety include Factory Mutual (FM), Underwriters Laboratory (UL), and Mine Safety and Health Administration (MSHA)
- Although equipment accuracy may be certified within set limits by the manufacturer or the National Institute of Occupational Safety and Health (NIOSH), whenever possible, equipment should be calibrated before and after use. For some instruments calibration in the laboratory may be acceptable and will save time and inconvenience in the field
- Operators should be thoroughly familiar with the instrument and operating instructions before use. Always read or review the manual prior to using an instrument in the field.

Advantages include:

- Immediate readout
- Portability.

Disadvantages include:

- Response usually is not specific to a single chemical
- Detection limits are generally in the range of acute health effects (i.e., field instruments may not have the sensitivity needed to measure low concentrations of pollutants)
- Relatively expensive compared with sample collection equipment
- Quality control can be difficult because it is hard to run blanks and calibration samples in the field
- Changes in temperature and humidity may affect the sensitivity and accuracy of the instruments.

Field monitoring instruments are widely used for air monitoring in emergency situations where it is important to know whether the air is safe to breathe or whether a respirator will have to be worn. They are also widely used for screening to determine approximate concentration levels and the number and placement of other types of samplers. The following sections describe the common types of instruments used to monitor various air contaminants.

11.8.1. Detection of Volatile Organic Chemicals (VOCs)

VOCs include components of natural gas, gasoline, and solvents. These chemicals can evaporate to form an explosive mixture in air and may be toxic at lower concentrations. The following analytical instruments have been developed as portable devices, which can be taken into the field for air monitoring. Each instrument is calibrated by determining the response of the detector to clean (unpolluted) air and to air containing a known concentration of a VOC. When the instrument is used to monitor polluted air, instrument calibration should depend on the type and concentration of VOCs present.

11.8.1.1. Photo Ionization Detector (PID)

A PID contains a miniature lamp, which emits a very short wavelength ultraviolet radiation. Inside the instrument, a small air pump continuously draws ambient air through a tiny chamber, which is flooded with ultraviolet light (See Figure 11-2). If the radiated energy is higher than the ionization potential of a particular organic chemical in the sample, the chemical will be ionized. The chamber also contains two electrodes charged to a high voltage and attached to a sensitive current measuring device. When ions are formed, electric current flows between the electrodes in proportion to the number of ions in the air. The instrument display generally has units of parts-per-million (ppm). This concentration should be interpreted as the equivalent response to the calibration gas, not the ppm of unknown VOCs in the air, as different chemicals have different responses to the instrument.

Advantages include:

- ▪ Stable, calibration remains fairly constant
- ▪ Easier to ship, no flammable gas is required

- Sensitive to commonly encountered VOCs
- Not affected by presence of methane in air.

Disadvantages include:

- Sensitive to high humidity
- Can have problems when used in below freezing temperatures
- Bulb must be cleaned occasionally
- Response to various chemicals depends on the photoionization potential of the chemical and type of bulb used
- Detection limit generally 2 or 3 ppm VOCs (higher for VOCs not easily ionized by UV light)
- Not specific to a single chemical.

11.8.1.2. Flame Ionization Detector (FID)

A FID works by burning contaminated air in a hydrogen flame within the instrument. The residue of most burned organic vapors contains positively charged carbon-containing ions, which are attracted to the negative collecting electrode in the chamber. As the ions are collected, a current is generated, which corresponds to the concentration of vapor present. The amount of current is shown on the instrument display.

Advantages include:

- Response is more consistent for differing chemicals than with the PID
- Less sensitive to moisture than the PID

Disadvantages include:

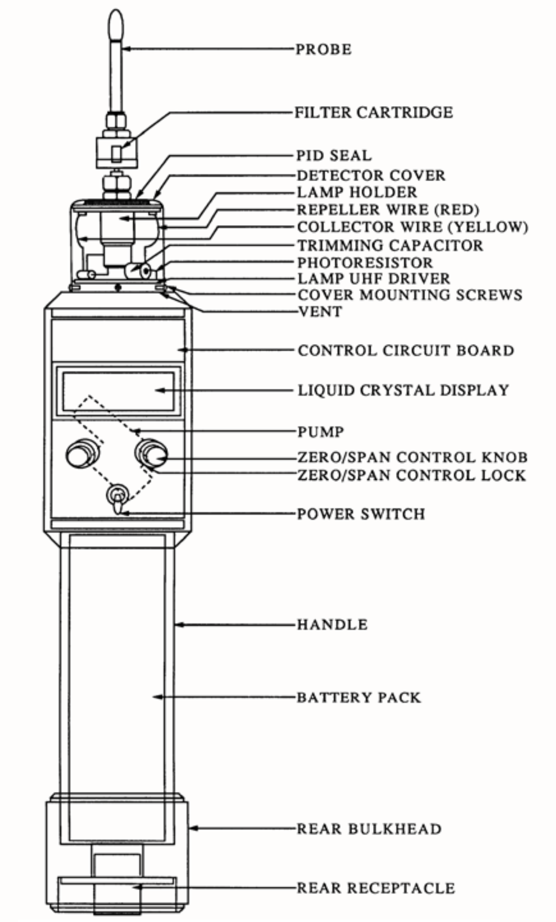

Figure 11-2 Photo Ionization Detector

- Need hydrogen for flame
- Sensitive to methane, which can mask the presence of toxic VOCs. Special methane filters are available
- Detection limit generally 2 or 3 ppm VOCs in air

11.8.1.3. Portable Gas Chromatograph (GC) and Gas Chromatograph/Mass Spectrometer (GC/MS)

A portable GC adds the benefit of separating mixtures of chemicals in an air sample. The air to be analyzed can be collected as either a whole air sample or in some instruments contaminants can be adsorbed on collection media and then desorbed to the instrument. Contaminants travel through a chromatography column and are separated based on their physicochemical properties, finally reaching a detector that outputs a signal that is proportional to the concentration of the contaminant. Common GC detectors utilize the same technology as the PID and FID.

A GC/MS utilizes a mass selective detector that provides structural information on the detected contaminant (mass spectrum).

Advantages include:

- Selectivity, contaminants are separated and can be identified by both the time they spend traveling through the chromatography column and by their mass spectrum
- Sensitivity, these instruments are generally more sensitive than a PID or FID

Disadvantages include:

- Powering the instruments may necessitate a portable generator or large batteries
- GC and GC/MS can be more difficult to operate and require more extensive training than the handheld instruments.

11.8.1.4. Infrared Radiation Absorbance Detector

Although VOC vapors are transparent to visible light, they will absorb infrared radiation. This detector passes a beam of infrared radiation through air. The extent to which the different wavelengths of infrared radiation are absorbed depends on the chemical present and its concentration. Because different chemicals have different absorbance spectra, the infrared radiation detector can be used to selectively measure specific chemicals. Some analyzers contain a library of calibration data for common gases and vapors, and have the capability to store data for multi user-calibrated gases.

Advantages include:

- Specific for particular chemicals, since different VOCs have different infrared radiation absorbance characteristics.

Disadvantages include:

- Detection limits are generally greater than those for the PID or FID.

11.8.1.5. Indicator Tubes

Indicator tubes are small glass tubes filled with a porous material coated with a reagent. When air is drawn through the tube, the chemical of interest reacts with the reagent, resulting in a color change. The length of filler that changes color depends on the concentration of the chemical of interest. Indicator tubes are available for both volatile organic and inorganic chemicals such as ammonia, hydrogen sulfide, sulfur dioxide, nitrogen dioxide, etc. Indicator tubes are available for use with hand-operated pumps (which draw the required amount of air through the tube in a minute or less), battery-operated pumps (which draw the required amount of air through the tube over an 8 hour period to provide a measurement of average air concentration), and diffusion tubes (which depend on the diffusion of the chemical into the tube over an 8 hour period).

Advantages include:

- Inexpensive
- Direct readout.

Disadvantages include:

- Sensitivity limited to concentrations of chemicals that cause acute symptoms, hence not sensitive enough for environmental sampling where chronic effects are important
- Limited accuracy (±20 – 50%)
- Not completely selective: Although tubes are designed to detect single chemicals, other chemicals present in the air may affect readings
- Wet (foggy) weather and low temperatures can affect reading accuracy
- Limited shelf life: Each sampling tube must be used within a few months to a year of purchase, depending on tube type.

11.8.2. Explosimeter

Explosimeters operate by passing an electric current through a platinum wire, causing it to heat up and glow. As air is passed over the hot wire, any VOCs in the air are oxidized on the surface of the wire. Oxidation increases the temperature and electrical resistance of the wire, resulting in a decrease in the current.

Advantages include:

- Rugged
- Sensitivity to VOCs is in the percent range, which is relevant to explosive mixtures.

Disadvantages include:

- Does not work if the concentration of oxygen in the air is abnormal; most explosimeters include an oxygen meter to assure that oxygen is present at proper level

- Does not work if hydrogen sulfide (H_2S) in air greatly reduces the meter sensitivity; most explosimeters also include a H_2S meter
- Not sensitive to the presence of VOCs at low levels that may still present toxicity hazards.

11.8.3. Detection of Hydrogen Sulfide (H_2S)

H_2S is a toxic volatile gas with a permissible exposure limit of 10 ppm in air. Though hydrogen sulfide has a strong odor of rotten eggs, exposure to it for more than a few minutes desensitizes the nose, so the odor cannot be used as a reliable indicator of the presence of hazardous concentrations. A portable H_2S meter has an electrochemical detector designed specifically to react to the gas. The meter is calibrated by using air containing a known concentration of H_2S.

11.8.4. Detection of Carbon Monoxide (CO)

CO is a colorless and odorless toxic gas. Portable CO detectors have an electro-chemical cell, which reacts specifically to CO. Combination meters with sensors for explosive gas mixtures, oxygen, hydrogen sulfide, and CO are available. Such meters are commonly used to check air quality in confined spaces prior to entry. CO can also be measured by non-dispersive infrared spectroscopy.

11.8.5. Particulates

The Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for particulates not otherwise regulated (3.5 - 100 µm in diameter) is 15 mg/m^3. Portable meters are available that work by passing a stream of air through a light beam and measuring the amount of light bouncing off particles present. The resulting measurement indicates the concentration of particles in the air.

Advantages include:

- Real time data
- Portable

Disadvantages include:

- Sensitive to fog and other aerosols
- Not size specific for dust particles
- Not easily calibrated in the field

11.8.6. Mercury Vapor

Mercury vapor detectors are used primarily to provide guidance on whether respirators are needed in areas where mercury has been spilled. Detectors work by exposing a gold foil to a flow of contaminated air. Mercury vapor present in the air is absorbed by the foil, changing its electrical resistance. Approximately 40 ten-second samples containing 100 µg Hg/m^3 may be taken before the sensor reaches saturation. At that point, a ten-minute heating cycle is manually activated to clear the accumulated mercury from the foil.

Advantages include:

- Specific to mercury
- Portable
- Real-time data (response time one to ten seconds)
- Detection limits adequate for work place monitoring (one to ten µg/m^3).

Disadvantages include:

- Not easily calibrated
- Quality control samples such as a blanks and spikes are difficult in the field
- Detects only atomic mercury fumes, not organic mercury compounds, which are even more toxic.

11.9. Collecting Samples for Laboratory Analysis

Air sampling procedures have been developed to enable representative samples of air or contaminant(s) of interest to be delivered to the laboratory for analysis.

11.9.1. Whole Air Samples

Collection of whole air samples is the simplest sampling technique. For this method, a sample of air is collected in a bag, jar, or canister, and the container is delivered to the laboratory.

A major concern with this type of air sample is the loss of the chemical of interest due to adsorption on or permeation through the container walls. Chemical reactions with the container walls and photochemical reactions induced by sunlight are also a concern for some compounds. It is important that the holding times be minimized and that the samples be analyzed as soon as possible. For very reactive or low concentration components this is not a suitable method.

At very high concentrations (e.g., several ppm), condensation may be a problem. Due to these limitations, this method should be used only for very stable volatile and semi-volatile organic chemicals such as hydrocarbons and chlorinated hydrocarbons with boiling points less than 150° C.

The following sections describe procedures, which may be used to collect whole air samples.

11.9.1.1. Bag Samples

Flexible bags for the collection of whole air samples are available in several different plastic materials including Tedlar®, Teflon®, Mylar®, and Saran®. Bag sampling is very useful in emergency situations where light weight and simplicity are an advantage and where the holding time will be very short.

Advantages include:

- Light weight
- Easily transportable
- Inexpensive.

Disadvantages include:

- Short holding times: Leakage and/or permeation of chemicals into and out of the bag can be a problem. Therefore, flexible bags should not be used for sampling for very low levels of air pollutants. Flexible bags are generally used to collect grab air samples to be analyzed within a few hours
- Cleanliness: Even new bags can be a source of contamination. Their cleanliness should be verified by blank analysis prior to use. Bags to be reused must be thoroughly cleaned by multiple evacuations and fillings with clean air. Cleanliness of each bag should then be verified before use. Cleanliness requirements depend on the concentration of the chemical to be measured and the analytical method. Reuse of flexible bags is not recommended when sampling for very low concentrations of organics
- Complicated field procedures: Additional equipment is required to fill flexible bags, including a vacuum pump and a hard-sided sampling box. An empty bag is put into the box and the inlet of the bag is attached to the sampling port of the box. The valve on the bag is then opened and the box is sealed. The vacuum pump is used to pull air out of the box, which reduces the pressure around the bag, causing it to fill with ambient air. The box is then opened and the valve on the bag is closed. The bag is then removed from the box, labeled, and delivered to the laboratory
- Transparent: Exposure to sunlight may cause photo-induced reactions of the chemicals in the air. Must store in darkness (e.g., wrap in black plastic bags)
- Reactivity: Some polymeric bag materials are reactive with certain compounds.

11.9.1.2. Glass Globe Samplers

Evacuated glass bulbs have been used extensively to collect whole air samples.

Advantages include:

- Ease: Simple to use when collecting grab samples; just open the valve to let air into the globe, close the valve, and deliver globe to the laboratory
- Cleanliness: Easily cleaned in the laboratory; should remain clean in storage
- Holding times: Neither absorption nor leakage is a significant problem, so holding times are longer than with flexible plastic bags.

Disadvantages include:

- Weight: Heavier than plastic bags
- Fragile: Subject to breakage in use and transportation
- Transparent: Exposure to sunlight may cause photo-induced reactions of the chemicals in the air; must store in darkness (e.g., wrap in black plastic bags)
- Reactivity: Surfaces can be reactive to some compounds.

11.9.1.3. Metal Canister Samplers

Stainless steel canisters, made less reactive by depositing a pure chrome-nickel oxide on the interior surface (Summa® polishing process), offer many advantages for collecting whole air samples. In general, a canister is evacuated to a

vacuum of approximately 30 inches of Hg. When opened to ambient air, the differential pressure causes air to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours) taken through a flow-restrictive inlet. A mass flow controller can maintain a constant flow rate from full vacuum to within about five inches of Hg below ambient pressure. With a critical orifice flow restrictor, a decrease in the flow rate occurs as atmospheric pressure approaches.
Advantages include:

- Holding time: Canisters are not subject to sample permeation or photo-induced chemical effects. (Sample integrity of trace level organic compounds stored in Summa® polished canisters has been demonstrated for many chemicals for up to 30 days.)
- Reusable: Can be reused after cleanup
- Cleanliness: Generally, does not absorb contaminants, so it is relatively easily cleaned. However, canisters used for high concentration samples should not be reused for very low concentration sampling. New canisters should be checked for cleanliness before use
- Rugged: Not readily breakable
- Sampling period: Can be used to collect either grab samples or samples over a 24 hour period
- Available in larger sizes than glass globes.

Disadvantages include:

- Heavy
- Expensive.

11.9.2. Air Component Samples

Another method of sampling air is to selectively remove the chemicals of interest from a measured volume of air and submit the concentrated sample to the laboratory for analysis. When obtaining any sample by selectively removing and concentrating the components of interest from a large volume of air, it is extremely important to remember that other undesirable things may also be concentrated such as background artifacts and water vapor. Because these undesirables may make subsequent analyses difficult or impossible, every effort should be made at the sampling stage to eliminate or minimize their inclusion. The following sections describe some of the sample concentration methods commonly used. The following section explains some methods used to collect and concentrate organic components from ambient air.

11.9.2.1. Passive Sampling Badges

A passive sampling badge consists of a solid adsorbent material covered with a membrane or plastic film. The chemicals of interest diffuse through the membrane and are adsorbed by the material inside the badge. The sampling badge is then put into a non-permeable (foil lined) pouch or plastic container and sent to a laboratory for analysis. At the laboratory, the adsorbing material is treated with solvents or heat to release the absorbed chemicals of interest.
Advantages include:

- Simple: no pumps, hoses, or batteries required
- Portable: can be attached to a worker's collar to measure the actual air breathed
- Several types of badges are available for different chemicals:
  - Volatile organic chemicals
  - Formaldehyde – 0.1 to 5 ppm
  - Glutaraldehyde – 0.2 to 5 ppm
  - Mercury vapor – detection limit < 0.05 mg/m^3.

Disadvantages include:

- High detection threshold: Suitable for work place exposure studies where the decision criteria are based on acute health effects. (Not suitable for most ambient air sampling)
- Accuracy: ±25%
- Requires 8 hour sampling period
- Not suitable for sampling for semi-volatile organic chemicals.

11.9.2.2. Active Air Samplers

These samplers use a pump to pull ambient air through the collection media. Sampling pumps are classified as high flow (500 – 3,000 cm^3/min) or low flow (50 – 200 cm^3/min). Flow rates should be maintained within ±5% of set rate.

Pump features may include:

- Diaphragm or piston-type of pumping action
- Flow regulator
- Rotameter or strokes counter to measure flow rate or sample volume
- Pulse dampener to smooth flow rate
- Timing mechanisms
- Safety features for use in flammable or explosive atmospheres.

Various collection techniques are used to sample the chemicals of interest from the air.

Advantages include:

- Chemicals of interest are concentrated, resulting in improved detection limits
- Standard methods have been defined and validated
- Samples are collected over long periods to obtain data on average contaminant levels.

Disadvantages include:

- Air flow may change over time – need wind speed and direction data throughout sampling period
- Complex equipment is used, which increases the likelihood of sampling errors
- A source of power may be required for the air sampling pumps.

11.9.2.3. Solid Sorbents

Solid sorbents are the primary collection means used to sample volatile and semi-volatile organic chemical vapors from air. Typically, large volumes of air are passed through a series of cartridges containing an inert adsorbing material. The organic chemicals are adsorbed from the air onto the surface of the adsorbent material. The laboratory extracts the adsorbed chemicals by using solvents or heat.

Unfortunately, solid sorbents are not selective for individual chemicals. Since broad classes of chemicals are adsorbed, the material may become saturated before the end of the sampling period and fail to adsorb all of the chemicals of interest from the air. This potential problem is controlled by using several sorbent cartridges in series. If the analysis of the last cartridge does not find any of the chemical of interest, it was either not present in the air or completely adsorbed by the material in the preceding cartridges.

A wide variety of solid sorbent materials have been developed for air sampling.

11.9.2.3.1. Organic Polymers

- **Tenax®**. Tenax® resin (2,6-diphenylene oxide polymer) is used in air sampling for volatile organic chemicals with boiling points between 35° and 100° C (molecular weights ranging up to several hundred AMU). Use of Tenax® is specified in EPA Method TO-1 and in stack sampling Method 0030. Tenax® must be specially processed before use to remove interfering chemicals. The processing procedure is specified in Methods 0030 and 0031.
- **Amberlite® XAD-2**. XAD-2, used in air sampling for polynuclear aromatic hydrocarbons (PAHs), is specified in the following EPA sampling methods:
  - Method TO-13
  - Method 0010 (Modified Method 5 Sampling Train) for stack samples
  - Method 0020 for stack samples

  XAD-2 resin, as supplied by the manufacturer, is impregnated with a bicarbonate salt solution to inhibit microbial growth during storage. Both the salt solution and any residual extractable monomer and polymer species must be removed before the resin is used. Appendix A to EPA Method 0010 gives detailed procedures for preparing XAD-2 sorbent resin.
- **Thermosorb/N**. Thermosorb/N is used to sample for N-nitrosodimethylamine.
- **Polyurethane foam**. Polyurethane is used to sample semi-volatile chemicals such as pesticides, PCBs, and dioxins from large volumes of air, achieving a very low detection limit.

11.9.2.3.2. Inorganic Powders

Includes **silica gel, Porasil®, Florisil®, alumina, and inorganic molecular sieves.** These materials are considerably more polar than organic polymeric sorbents and thus capture polar compounds. However, the inorganic powders can become deactivated by adsorbing water moisture from the air. Silica gel will adsorb compounds such as alcohols and phenols and other oxygen-containing compounds such as esters, aldehydes, and ketones. Florisil® can be used to collect chlorinated pesticides and PCBs from air.

11.9.2.3.3. Carbon Adsorbents

Carbon materials are a much stronger adsorbent than organic polymers and therefore allow efficient collection of highly volatile organic contaminants such as vinyl chloride, benzene, and toluene. However, the strong adsorption of carbon adsorbents can be a disadvantage in cases where recovery by thermal desorption of less volatile is desired because of the high temperatures required (e.g., 400° C).

There are a variety of carbon based adsorbents available with widely varying adsorption properties. The commonly available classes of carbon adsorbents include:

- **Carbon Molecular Sieves**. Sold under trade names such as **Spherocarb®**, **Carbosphere®**, **Carbosieve®**, and **Ambersorb® XE-347**, these materials have been used to sample air for very volatile organic chemicals such as vinyl chloride, ethylene dichloride, and methylene chloride.
- **Carbonaceous polymeric adsorbents** are available under trade names **Ambersorb® XE-340, XE-347**, and **XE-348**. XE-340 appears to be useful for volatile compounds in the C_4 to C_6 boiling point range, a volatility range not covered by organic polymeric adsorbents such as Tenax®.
- **Activated carbon**. Conventional activated carbons have a porous structure, which leads to difficulty in recovering adsorbed materials. Therefore, this material is rarely used as a sampling media.

11.9.2.4. Chemical Derivitization Techniques

Ambient air can be exposed to a chemical reagent that reacts with the volatile organic chemical of interest, forming a non-volatile product trapped in the sampling device. Integrated sample collection and derivitization of carbonyls with 2,4-dinitrophenyl hydrazine (DNPH) is a particularly good example of the utility of concentration and derivitization methodology.

The reagent can be in solution in a liquid impinger or "bubbler" or can be adsorbed on the surface of a porous solid. Liquid impingers require considerable glassware that must be specially cleaned, and the liquids must be handled in the field under difficult conditions. In addition, the impinger may have to be immersed in a bath of ice water to prevent the evaporation of the reagent-containing liquid.

Cartridges of solid sorbents coated with derivitizing reagent can provide much higher collection efficiencies than a liquid impinger. A variety of sorbent media can be used including: silica gel, glass beads, glass fiber filters, Amberlite® XAD-2 resin, Florisil®, Chromosorb P®, Carbopak B®, and C18 coated silica. Although the solid phase method is much superior to the impinger method, the sampling is not without problems such as:

- incomplete collection
- reactivity of carbonyls
- surface adsorption of carbonyls
- artifact and interfering compound formation
- blank contamination
- variable rates of formation of derivatives for various compounds
- interference of ozone and sulfur dioxide

11.9.2.5. Nebulization/Reflux Concentration

This method is a modification of the bubbler/impinger methodology. In this case, instead of bubbling air sample through a liquid scrubbing solution, the liquid is nebulized to a fine mist inside a glass bulb. Air is introduced through a small orifice located in close proximity to the nebulization tip and analytes of interest in the air flowing through the mist are transferred to the mist droplets. In the fine mist state, the surface area for analyte contact is greatly increased, resulting in significantly improved collection efficiencies. A non-wettable filter (usually Teflon®) collects and coalesces the mist back into the bulb while passing scrubbed air out. The liquid scrubber solution can either dissolve the analytes of interest or chemically derivatize them.

Table 11-1 lists the EPA standard methods for sampling ambient air and their respective collection media.

Table 11-1 EPA Standard Air Contaminant Sampling Methods for Toxic Organic Compounds

Method	Description	Types of Compounds Determined
TO-1	Tenax® GC adsorption and Gas Chromatography/Mass Spectrometry (GC/MS) analysis	Volatile, nonpolar organics (e.g., aromatic hydrocarbons, chlorinated hydrocarbons having boiling points in the range of 80° to 200° C
TO-2	Carbon molecular sieve adsorption and GC/MS analysis	Highly volatile, nonpolar organics (e.g., vinyl chloride, vinylidene chloride, benzene, toluene) having boiling points in the range of -15° to +120° C
TO-3	Cryogenic trapping and GC/FID or ECD analysis	Volatile, nonpolar organics having boiling points in the range of 10° to 200° C
TO-4	High volume PUF sampling and GC/ECD analysis	Organochlorine Pesticides and PCBs
TO-5	Dinitrophenylhydrazine (DNPH) liquid impinger sampling and HPLC/UV analysis	Aldehydes and Ketones
TO-6	Aniline liquid impinger with High Performance Liquid Chromatography (HPLC)	Phosgene
TO-7	Thermosorb/N adsorption	N-Nitrosodimethylamine
TO-8	Sodium hydroxide liquid impinger with High Performance Liquid Chromatography (HPLC)	Cresol, Phenol
TO-9	High volume Polyurethane Foam (PUF) sampling with High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)	Dioxins (polychlorinated dibenzo-p-dioxins), Furans (polychlorinated dibenzofurans)
TO-10	Low volume Polyurethane Foam (PUF) sampling with Gas Chromatography/Electron Capture Detector (GC/ECD)	Pesticides
TO-11	DNPH coated adsorbent cartridge followed by High Performance Liquid Chromatography (HPLC) detection	Formaldehyde
TO-12	Cryogenic Preconcentration and Direct Flame Ionization Detection (PDFID)	Non-Methane Organic Compounds (NMOC)
TO-13	PUF/XAD-2 adsorption with GC and HPLC detection	Polynuclear Aromatic Hydrocarbons (PAHs)
TO-14	Summa Canister collection with GC Analysis	Volatile Organic Compounds
TO-15	Summa Canister collection with GC/MS Analysis	Volatile Organic Compounds

11.10. Particulates

Many toxic chemicals present in ambient air are associated with solid particulates or aerosols. Organic chemicals, such as semi-volatile organics and polar compounds, may adsorb to particulates or condense to varying degrees and exist in a complex dynamic solid-vapor equilibrium with particulate matter. Volatile metals, such as cadmium and lead, in stack gases may condense on the surface of particulates. Some toxic compounds such as asbestos are present in ambient air only as particulates. Air sampling for particulates uses filtration, centrifugation, or impaction to remove particles from the air (See Figure 11-3 for an example of a typical particulate sampler). Certain adsorbed chemicals may evaporate off the surface of the particles during the capture process. Therefore, there is often the need for a gas phase collection device (e.g., adsorbent) downstream from the particulate capturing device. Analysis of both collection media would be necessary to determine the total amount of chemical present in the ambient air, but the results may not describe the actual phase distribution of the chemical in the air.

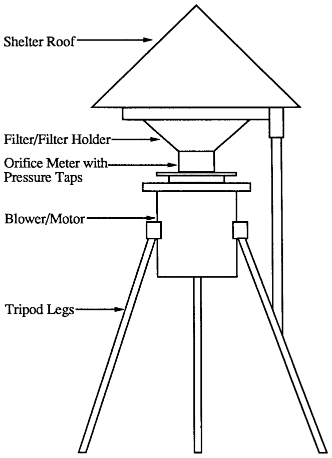

Figure 11-3 Particulate Sampler

11.10.1. Filtration

The most common method for collecting particulates is filtration. Many filter media with different filtration characteristics are available. The most commonly used media are:

- **Cellulose fiber filters** (e.g., Whatman No. 41 filter paper)
- **Glass or quartz fiber filters** withstand higher temperatures, are less affected by moisture in the air, and have higher collection efficiencies than cellulose filters. However, they have less strength and are more expensive than cellulose filters.
- **Membrane filters** (cellulose esters, Teflon®, nylon, etc.) have a well-defined pore size, but are limited to low flow-rate sampling. Many membrane filters will dissolve in solvents. This may be an advantage or disadvantage depending on the chemicals of interest.
- **Nucleopore filters** have well-defined pore sizes but are expensive and are limited to low volume sampling.

A variation on fiber filters is the thimble filter, which uses a filter paper thimble filled with fluffed cotton. For high temperature gases, an aluminum thimble replaces the paper thimble.

11.10.2. Centrifugal Collection and Impaction

EPA ambient air standards for particulates are based on the concentration of particles 10 µm in diameter or smaller. Methods for collecting size-fractionated particulate samples generally use cyclones (centrifugal collectors) or impactors. Both of these devices collect particles of a specific size range based on the design of the apparatus. Cyclones tend to have a rather broad size cut; impactors have a much sharper size cut.

- **Cyclone Collectors.** The cyclone is one of the most common types of solid particle collectors. It is an inertial, centrifugal separation, which separates particles by whirling them in a cyclonic motion in a conical shaped collection device. Particulates strike the wall of the collector and are collected at the bottom of the collection device.
- **Impactors/Impingers.** Impactor-type collectors function based on the different momentum of gases and particulates in a flowing stream. The separation is achieved by forcing the stream onto a solid surface where a specific size fraction of the aerosol is deposited. The solid surfaces may be a

baffled chamber, slotted plate, channels, or other obstructive arrangement.

11.11. Stationary Source Emission Samples

Stack, vent, and duct samples are collected to evaluate the concentration of hazardous chemicals and particulates emitted into the air. Emission testing may be done to:

- Comply with a set of regulatory requirements
- Provide emission test data for a regulatory request
- Collect information for control equipment design specifications
- Determine control equipment efficiencies
- Acquire emission inventory data for historical, contaminant reduction, or process control purposes.

Sources may range from large stacks to small vents. Sources can include:

- Process gas streams
- Control device inlet ducts
- Area ventilation sources
- Storage tank vents
- Material handling systems
- Surface impoundments and landfills
- Pumps, valves, flanges, and other devices with seals.

Basic sample collection principles are the same as those used for ambient air. However, the high velocity and high temperature of stack gases and some other stationary emission sources requires that special procedures be used to obtain representative samples.

Many stationary emission sources can now be monitored continuously rather that by discrete samples. **Continuous Emission Monitoring Systems (CEMS)** can collect more data than discrete samples, and digitized dataloggers can provide a variety of sampling time intervals.

For any stationary source monitoring program, end use of data, data quality objectives, and QC requirements must be considered. Frequently, a screening test may be needed to determine the long range testing plan. All monitoring and measuring equipment must be kept in good working order and must be calibrated to traceable standards where applicable.

11.11.1. Special Problems with Stack Vent and Duct Sampling

Collecting samples from stack emissions requires procedures to address the following problems:

- **Non-Uniform Conditions Across Stack Vent or Duct.** One cannot assume that the stack gas is the same in the center of the stack, vent, or duct as it is near its walls. Therefore, the gas must be collected at various locations across the source to ensure the sample is representative of the average conditions in the source.
- **Velocity Effects at Sampling Orifice.** The tip of the sampling probe or nozzle can cause turbulence in the stack, vent, or duct gas that can deflect particles away from the probe, resulting in an invalid sample. It is important that the flow rate and flow direction of the sampled gas passing through the probe nozzle tip opening be the same as those in the source in order to minimize the turbulence caused by changes in gas flow at the tip of the probe. The gas flow rate and flow direction through the sampling probe or nozzle tip should be equal to gas flow rate and flow direction of gas passing through the source (i.e., isokinetic).
- **Condensation in Sampling Probe.** High temperature gas in a stack, vent, or duct may contain concentrations of water and chemicals that could condense out before reaching the sampling equipment if the gas is allowed to cool in the sampling probe. Therefore, precautions must be taken to maintain the temperature of the gas in the probe, while cooling the gas and condensing out the moisture as necessary in the sampling equipment to ensure sampling effectiveness. Temperature in the sampling line must be held above the dew point of the normally gaseous components.
- **Corrosivity of Stack Gases.** Since stack gases are frequently corrosive, sampling devices must be made of non-corrosive materials.
- **Collector Efficiency.** Generally, collectors should be at least 95% efficient unless the

reproducibility of samples is very high and representative of the gas stream.

- **Sample Collector Orientation.** The long axis of the sampling head must be parallel to the direction of gas flow.
- **Total Gas Flow.** For accurate quantitation, the total gas flow must be determined. Volume of gas flow is usually determined by traversing the cross section of the stack, vent, or duct using a Pitot tube to obtain gas velocity. Velocities calculated from the Pitot tube readings are averaged and multiplied by the cross sectional area of the source to obtain the gas volume.

11.11.2. Specified Procedures

In order to ensure the accuracy of reported stack, vent, or duct sampling data, the EPA specifies the sampling procedures in great detail. The sampling equipment is available from a number of commercial suppliers, and the methods should be followed precisely. The following sections summarize stack sampling methods that the EPA has developed for hazardous waste incinerators.

11.11.2.1. Method 0010 – Modified Method 5 Sampling Train

Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. Principal components of the train include a high efficiency glass or quartz fiber filter and a packed bed of porous polymeric adsorbent resin **(XAD-2)**. The filter is used to collect organic-laden particulate materials and the porous polymeric resin to adsorb semi-volatile organic species. Commercial models of this equipment are available.

11.11.2.2. Method 0020 – Source Assessment Sampling System (SASS)

Particulate and semi-volatile organic materials are extracted from a source at a constant rate near isokinetic conditions and are collected in a multi-component sampling train. Three heated cyclones and a heated high-efficiency fiber filter remove and collect the particulate material from the sample and a cartridge containing 150 grams of XAD-2 resin.

11.11.2.3. Method 0030 – Volatile Organic Sampling Train (VOST)

This method collects a 20-liter sample of stack gas containing volatile principle organic hazardous compounds (POHCs). The stack gas is collected at a flow rate of 1 liter per minute using a glass-lined probe and a VOST. The gas stream is cooled to 20° C by passage through a water-cooled condenser. Volatile POHCs are collected on a pair of sorbent resin traps. Liquid condensate is collected in an impinger placed between the two resin traps. The first resin trap (front trap) contains approximately 1.6 grams of Tenax® and the second trap (back trap) contains approximately 1 gram each of Tenax® and petroleum-based charcoal, 3:1 by volume. The VOST is designed to be operated at 1 liter per minute with traps being replaced every 20 minutes for a total sampling time of two hours. Traps may be analyzed separately or combined into one trap to improve the detection limit. Recent experience has shown that when less than maximum detection ability is required, it is acceptable and probably preferable to operate the VOST at 0.5 liters per minute for a total of three 40 minute periods. This preserves the two hour sampling period, but reduces the number of cartridge changes in the field as well as the number of analyses required.

11.12. Measurement of Air Volume Sampled

To obtain the proper results, the total volume of air sampled must be known. The flow rate of air through the sampling media shall be measured at both the beginning and end of the sampling period because the resistance of air through the sampling media may increase as it is exposed to contaminated air, resulting in a decreased flow rate. The sampling device should also be checked periodically to make sure that the sampling pump is working. The air flow rate is the average of the beginning and end flow rates. If the sampling pump is connected to line power and does not contain a timer, a clock should be connected to the same outlet to measure the duration of sampling if the power is interrupted. The total amount of air that is sampled is calculated from the measured air flow rates and the sampling duration:

$$\textit{Total Volume} = \textit{Average Flow Rate} \times \textit{Time}$$

11.12.1. Low Flow Rate Samples

The most common type of air flow measuring device for low flow rate air samples is the soap film calibrator. This device is placed between the sampling device and the air pump, and a soap film is created across the end of the straight glass tube. This film acts as a frictionless piston, and moves up the tube as air is drawn through the calibrator. The time required to move the film past two points on the tube is either measured with a stop watch or is detected by photoelectric cells in the device.

Although this device is accurate, it is rather fragile and requires some practice to generate the soap films and to make the time measurements. The photoelectric cell detectors may not function in bright sunlight, so this type of device might have to be shaded with a dark cloth or plastic bag during use. After the air flow rate measurement is completed, the calibrator is disconnected from the pump and the sample collection device and the pump are connected directly to the sampling device.

The sample media used for low flow rate samples may consist of glass tubes that contain solid adsorbent media, midget impingers, or filter cassettes. The air is drawn through the collection media using a portable air sampling pump.

11.12.2. High Flow Rate Samples

Most high flow rate air sampling devices are equipped with a manometer or other flow rate indicator. The flow rate must be measured at the beginning and end of each sampling period and should be checked periodically during the sampling period. The total volume sampled is calculated by multiplying the average flow rate by the sampling time.

11.13. Hazard and Safety Precautions

The safety plan or Health and Safety Plan (HASP), as appropriate to the scope of the project, should address all anticipated hazards for each task. The following is a short list of more common (and commonly overlooked) hazards associated with air sampling activities.

11.13.1. Environmental Hazards

Safety precautions include a review of possible environmental hazards before entering the site and the use of proper clothing and equipment. Workers performing stack sampling and air monitoring during emergency situations may be exposed to hazardous levels of air pollutants. Therefore, the safety plan or HASP, as appropriate to the scope of the project, must specify what kind of real-time air monitoring will be performed, the action levels for the use of respirators, and the types of respirators to be worn if action levels are exceeded. If there is a chance sampling personnel may be exposed to hazardous air, planning should include:

- Staffing requirements for real-time air monitoring and personal protective equipment (PPE) support personnel
- Use and availability of real-time air monitoring equipment, including calibration standards
- Assignment of sampling personnel trained in the use of PPE and real-time air monitoring procedures, and medically approved for work in respirators.

11.13.2. Stack, Vent, and Duct Sampling Safety

Safety concerns are of critical importance in performing stack sampling due to the possibility of fume exposure, falling, dropping equipment on workers below, contact with hot surfaces, shock, and weather related hazards such as ice, snow, and rain.

Procedures developed for each sampling task should carefully address all relevant safety issues, such as criteria for use of PPE, fall protection lanyards, and heat resistant (Nomex®) coveralls and gloves.

11.13.3. Instrument Hazards

Gases used to calibrate and operate some instruments come in pressurized cylinders and many are flammable. Proper care should be taken when handling these materials. Light sources from some instruments can cause eye damage when viewed directly.

11.14. Preparation for Sampling

11.14.1. Role of the Sampler

The role of sampling personnel is critical to the overall conduct of an air sampling program. The role of sampling personnel is to:

- Prepare or review Sampling and Analysis Plan (SAP)
- Be familiar with all required sampling protocols
- Assemble and verify cleanliness and operational status of all required equipment
- Calibrate, standardize, and operate air quality monitors
- Collect air data
- Collect samples for specific emission activities
- Ensure samples are properly preserved and transported to laboratory within holding times
- Document and record all pertinent information

11.14.2. Guidelines for Air Sampling

The SAP should specify when, where, and how air samples are to be collected. Careful and complete planning is required to ensure needed equipment and trained personnel are available to perform the method of sampling needed to collect the desired data. The following procedures should be followed when mobilizing for an air sampling program:

1. Review SAP and the safety plan or HASP. Identify special equipment and associated security requirements.

2. Identify sampling locations and determine whether each presents a problem for access, security, or availability of required utilities.

3. Prepare a staffing plan, which includes a description of the responsibilities and required training for each of the sampling personnel. For air samples collected over a long duration (8 to 24 hours), determine how many sampling personnel are needed to monitor air flows and equipment performance and how many security personnel are needed to ensure that the sampling equipment is not tampered with while operating.

4. Coordinate planning and scheduling with facility personnel to ensure access to sampling locations and availability of security personnel, utilities, respirator support personnel, and emergency support services.

5. Coordinate with the laboratory to ensure availability of its services within holding time tolerances. The laboratory may be able to provide significant support in preparing or furnishing sampling equipment and shipping containers.

6. Procure required sampling equipment, including:

- Any required calibration standards
- Additional sampling devices as required for blanks and duplicates and to allow for field breakage
- Check sampling canisters to ensure vacuum has been maintained
- Check all battery powered pumps to ensure batteries are charged and will hold a charge
- Check air flow measuring equipment to ensure operability.

7. Procure all required PPE, including respirators and correct cartridges, real-time air monitoring equipment, and fall protection equipment (for stack sampling).

8. Check availability of weather data. It may be necessary to install a portable weather station at the sampling site to obtain a record of wind speed and direction, temperature, humidity, air pressure, and precipitation events.

9. Ensure provisions are in place to monitor and record events that occur at the sampling location, which might affect samples. This includes any activities operating in close proximity, which may affect sample integrity. It is important that all unusual releases occurring while sampling be thoroughly documented as to time, location, duration, and types and quantity of chemicals released to the atmosphere. Releases can include spills and an unusual amount of dust from facility operations or construction activities.

10. Check the Field Sampling Forms to be sure that unique sample numbers have been assigned to each sample.

11. Obtain a Field Log Book/Field Notes. Keep air-monitoring field notes neat and legible in a

legally defensible form. When sampling begins, document the start time, flow rate, weather conditions (ambient temperature, barometric pressure, humidity, etc.), locations of sampling equipment, and other pertinent information in the Field Log Book/Field Notes. During sampling, check the weather conditions frequently and enter them in the Log Book. Check the sampling equipment periodically. Enter findings (e.g., sampler off, knocked over, etc.) in the Log Book. If, at any time, the sampling equipment must be moved, note the time of the movement, the new locations, and the reasons for movement. When sampling is completed, record the time and ending flow rate. When using a multiple part form, place part of the form in the Log Book per the SAP or SOPs. Include calculations (such as the volume of air sampled, average flow rates, etc.) and pertinent observations.

12. Ensure that all materials required for sampling are available in sufficient quantities for the planned work.

13. Shipping: Sample jars and cartridges must be packed with bubble wrap, styrofoam, or other cushioning material to prevent breakage during shipment to and from the site. Vermiculite contains asbestos and shall not be used.

The validity of air samples depends on care taken to comply with the detailed sampling procedures, which includes equipment calibration, air flow measurements, and the collection of weather data. Variables such as the types of chemicals present in the air, on-site conditions and activities, and weather conditions can greatly affect sampling performance.

If the SAP specifies a standard EPA procedure, a complete copy of the procedure should be obtained and reviewed to ensure necessary equipment is available.

Specified steps of the EPA procedure should be followed as closely as possible. Any deviations from the procedure should be noted in detail in the Field Log Book/Field Notes

Chapter 12. Process Sampling

12.1. Purpose

This chapter discusses the need and provides strategies for sampling associated with remediation technologies.

12.2. Scope

Sampling of water (and vapors) is commonly conducted in groundwater treatment plants to support decisions regarding compliance with discharge permits and for making operational decisions at the plant. Sampling may occur for plant influent and effluent and intermediate samples may be taken before and after specific processes in the treatment train to:

- Assess adequacy of performance
- Tailor the addition of chemical amendments
- Monitor equipment operation.

For example, the need to replace reactive media (such as granular activated carbon (GAC)) is based on the above-described process monitoring. Sampling is usually a simple matter of collecting the necessary volume directly in the sample container from a valved sample tap. Purging is usually not required. Analysis of samples is typically conducted by off-site fixed laboratories, although at a few sites frequent sampling may justify an on-site laboratory, particularly during the early phases of plant start-up and operation. Few plants need on-site labs following the first year or two. In some cases, particularly where the compliance with standards is critical, auto-sampler or in-line sensor devices are used to automate the sampling and analysis process and to provide near-real-time data for process control, including plant shut down. In-line sensors are commonly used to monitor pH, and to control chemical feed pumps for pH adjustment. Auto-samplers are often used for collection of composite samples (e.g., 24-hour composite). Composite sampling, in conjunction with off-site analysis, is often required for effluent monitoring of contaminant levels, and for effluent toxicity testing. Composite sampling is generally not applicable for VOCs. All sampling should be conducted in accordance with the project SAP, or applicable permit.

A brief discussion of process sampling considerations is shown below for only the most common treatment technologies for remediation of contaminated groundwater and soils. The discussion is limited to the following treatment technologies.

12.3. Ex situ Groundwater Treatment

12.3.1. Liquid-Phase Granular Activated Carbon

Sampling is usually performed at the influent and effluent of the vessels containing the liquid phase GAC. Usually there will be a set of two vessels, operated in series (i.e., lead / lag configuration). Typically, samples are collected from the influent of the lead vessel, from a port between the lead and lag vessels, and from the effluent of the lag vessel. The need to replace adsorptive media, such as liquid phase GAC, is based on the above-described process monitoring.

Sampling is usually a simple matter of collecting the necessary volume directly in the sample container from a valved sample tap. Purging is usually not required. Analysis of samples is typically conducted by off-site fixed laboratories, although at a few sites frequent sampling may justify an on-site laboratory, particularly during the early phases of plant start-up and operation. Please see the following references for additional information:

- USACE DG 1110-1-2 for additional discussion of sampling approaches
- UFGS 43 31 13.14 (11225), Downflow Liquid Activated Carbon Adsorption Units.

12.3.2. Air Stripping

Because air stripping results in some of the contaminants (i.e., VOCs) being transferred into the vapor phase, monitoring of both water and air is often required. The performance (i.e., removal efficiency) of the air stripper is monitored by collecting water samples from ports positioned before and after the air stripper.

During air stripping, VOCs are transferred from the liquid to the vapor phase. A sample port on the air-discharge line of the air stripper is usually available for collecting air samples to determine the concentration of contaminants being discharged into the air. If the air is being discharged directly to the atmosphere, then monitoring of the air discharge line may be required to demonstrate that regulatory air-discharge criteria are not being exceeded. In some instances, the air discharge from the air stripper must be treated before it can be released to the atmosphere. The most common treatment technology for removing VOCs from the air discharge from air strippers is vapor-phase GAC.

Because the air discharge from air strippers will be laden with moisture, it will typically have to be routed through an air-water separator (i.e., knock-out tank) before vapor-phase GAC treatment. The purpose of the air-water separator is to remove entrained water from the air stream. The air-water separator will usually include a tank for collection of water. The water that accumulates in the air-water separator constitutes another waste stream, which may require sampling. In some cases the water that accumulates in the air-water separator can be blended back into the untreated water stream (upstream from the air stripper influent), in which case sampling may not be required.

Use of in-line sensors for continuous monitoring of parameters such as pH is relatively common for groundwater treatment plants. An important side-effect of air stripping is that it often results in an increase in the pH of the water, after it passes through the air stripper. The pH may increase due to removal of dissolved carbon dioxide, and a shift in the equilibrium between carbon dioxide, carbonic acid, bicarbonate, and carbonate ion. If the pH becomes elevated, this often results in an increased propensity for precipitation of minerals (e.g., calcium carbonate). Mineral precipitation can lead to rapid fouling of the air stripper and coating of surfaces downstream from the air stripper. Chemical feed systems are often used to prevent, or slow the rate of fouling. Acids or sequestrants are often introduced upstream from the air stripper to control mineral precipitation. Acid-feed systems are usually equipped with in-line pH meters to control the rate of acid addition.

Please see the following references for additional information:

- USACE DG 1110-1-3 for additional discussion of sampling approaches
- UFGS 43 13 13.13 (11226), Vapor Phase Activated Carbon Adsorption Units.

12.4. In situ Groundwater Treatment

All of the processes described below represent alternatives for groundwater remediation. All of these processes rely on conventional groundwater monitoring methods, using monitoring wells. The primary parameters that require monitoring are the contaminant levels, and contaminant breakdown products (when applicable). The primary basis for monitoring the effectiveness of these treatment processes is demonstrating that contaminant levels (and breakdown products, if applicable), are decreasing (in response to treatment), at an acceptable rate, over time.

In addition to monitoring contaminant levels there are a host of other “indicator parameters” that may need to be included as monitoring parameters. The suite of indicator parameters is technology-specific, and will vary depending on the specific compounds that are being introduced into groundwater as part of the treatment process. Since the monitoring for in situ treatment will typically involve sampling of existing monitoring wells, and such sampling is covered elsewhere in the document, the focus in this section is on the typical analytical requirements for the various in situ technologies. A brief description of the technologies also are included.

12.4.1. Monitored Natural Attenuation (MNA)

For MNA, the primary parameters that require monitoring are the contaminant levels and contaminant breakdown products. The primary basis for monitoring the effectiveness of MNA is demonstrating that contaminant levels (and breakdown products, if applicable), are decreasing, at an acceptable rate, over time.

Measurements of organic carbon levels in groundwater (e.g., total organic carbon) are often used to help determine if naturally occurring, or anthropogenic, electron donors are present.

Groundwater samples are also commonly tested for levels of electron acceptors to determine if depletion of electron acceptors is occurring, and whether or not reductive dechlorination is contributing to attenuation of contaminants. In some cases, a relatively large suite of parameters may be necessary to provide the necessary lines-of-evidence to demonstrate the extent to which MNA is occurring, and to assess if attenuation processes are proceeding at a sufficient rate. Usually, the number of monitoring parameters and the frequency of monitoring can be reduced after a record of several years of groundwater monitoring data has been acquired and the trend in each of the parameters has been established. Refer to the references below for additional information on monitoring parameters and sampling approaches:

- Interstate Technology and Regulatory Cooperation (ITRC) Natural Attenuation of Chlorinated Solvents in Groundwater: Principles and Practices (September 1999)
- Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater**,** EPA 600-R-98-128, 1998.

12.4.2. Enhanced Anaerobic Bioremediation (EAB)

EAB usually involves injection of electron donor formulations into groundwater in order to stimulate anaerobic conditions, and to promote reductive dechlorination of chlorinated solvents. Electron donor formulations can include: corn syrup, molasses, emulsified oils, whey, chitin, etc. Introduction of electron donor results in depletion of electron acceptors. Microorganisms consume the most energetically favorable electron acceptors first (i.e., usually DO and nitrate), followed by less favorable electron acceptors such as sulfate. Chloroethenes, such as TCE, become available as electron acceptors after the more favorable electron acceptors have been largely depleted. In some instances, it may also be necessary to inject cultures of dechlorinating microorganisms (i.e., bioaugmentation), to promote reductive dechlorination.

The primary parameters that require monitoring are the contaminant levels and contaminant breakdown products. The primary basis for monitoring the effectiveness of these treatment processes is demonstrating that contaminant levels (and breakdown products, if applicable), are decreasing (in response to treatment), at an acceptable rate, over time.

Measurements of organic carbon levels in groundwater (e.g., total organic carbon) are often used to help determine the adequacy of delivery and distribution of electron donor. Groundwater samples are also commonly tested for levels of electron acceptors to determine if depletion of electron acceptors is occurring. Most of the same parameters that are used for MNA are also included as parameters for EAB. Refer to the below references for additional information on monitoring parameters and sampling approaches:

- Protocol for In Situ Bioremediation of Chlorinated Solvents Using Edible Oil (http://www.clu-in.org/download/remed/Final-Edible-Oil-Protocol-October-2007.pdf)
- Principles and Practices of Enhanced Anaerobic Bioremediation of Chlorinated Solvents.(http://costperformance.org/remediation/pdf/principles_and_practices_bioremediation.pdf)

Testing for the presence of genetic material from dechlorinating microorganisms (i.e., 16S ribosomal RNA (rRNA) from Dehalococcoides (DHC) organisms) and specific DHC reductive dehalogenase (RDase) genes is often performed to assess the microbial capacity for reductive dechlorination in groundwater. These gene-targeted molecular tools are used to assess the extent to which naturally-occurring DHC are present, or to assess how well the bioaugmentation culture has been distributed in groundwater. The number of copies of 16S rRNA DHC genes, and RDase genes can be determined via quantitative real-time polymerase chain reaction (qPCR) analysis. This testing is available thru commercially laboratories such as SiREM and Microbial Insights.

For more information on qPCR testing see the following reference:

- Kirsti M. Ritalahti, Benjamin K. Amos, Youlboong Sung, Qingzhong Wu, Stephen S. Koenigsberg, and Frank E. Löffler; Quantitative PCR Targeting 16S rRNA and Reductive Dehalogenase Genes Simultaneously Monitors Multiple Dehalococcoides Strains**;** Appl. Envir. Microbiol., April 2006; 72: 2765 – 2774.9.2.2 Chemical Oxidation.

This technology involves the injection or placement of some type of chemical oxidant into the subsurface. The reagents often include permanganate, stabilized hydrogen peroxide, persulfate, and ozone. The primary considerations will be verifying the efficacy of the distribution of the reagents to assure adequate contact with the contaminants. The analytical requirements would normally include monitoring of oxidation/reduction potential, preferably using in situ measurement with downhole instruments. There would also be monitoring of the reagents themselves or reaction products. Permanganate addition creates a purplish hue to groundwater so colorimetric analysis may be possible to assess distribution of that reagent. Monitoring of target contaminant in standard groundwater samples would be an integral part of the sampling and analysis program.

For additional information, please refer to the following reference:

- Technical and Regulatory Guidance for In Situ Chemical Oxidation of Contaminated Soil and Groundwater, Second Edition (January 2005).

For oxidation of contaminants using ozone, the monitoring of residual ozone may be required for safety reasons.

12.4.3. Aerobic Biodegradation via Oxygen-Releasing Chemicals

A number of products are commercially available that rely on chemicals for slow-release of oxygen into groundwater. These chemicals are used to supply DO to support aerobic biodegradation of hydrocarbons, such as benzene, toluene, ethylbenzene, and xylene (BTEX), in groundwater. The chemicals usually contain either magnesium- or calcium-peroxide based formulations that, when hydrated, gradually release DO into groundwater. Oxygen-release chemicals can be applied in a number of different ways. In dry form, the chemicals can be packed into “socks” that are suspended below the water table, in wells. The dry powder can also be spread across the surface at the bottom of excavated areas for treatment of residual dissolved-phase hydrocarbon contamination; wetting of the area may be accomplished by applying water during backfilling. Alternatively, the dry chemicals can be mixed with water to form a slurry. The slurried material can then be delivered to groundwater via injection into existing wells or by DPT. Injection of the slurry under pressure usually results in distribution over a greater area around the injection point (i.e., greater radius of influence) relative to passive delivery techniques such as the suspended “sock” method.

Hydrogen peroxide has also been used to supply DO, and promote aerobic biodegradation of organic contaminants. A solution of hydrogen peroxide can be mixed into water that is being injected, or the hydrogen peroxide solution can be slowly metered into a well to provide a steady supply of DO. The hydrogen peroxide will kill microorganisms in the immediate vicinity of the injection well, but it breaks down rapidly, and releases oxygen as the main byproduct. The concentration and flow rate of the hydrogen peroxide solution must be carefully controlled to limit inhibition of microbial activity to the immediate vicinity of the injection points.

The analytical requirements would include the monitoring of DO and oxidation-reduction potential in groundwater as an indication of the extent of chemical distribution. Such measurements are best made downhole using suitable probes or low-flow sampling techniques. Monitoring of target contaminants in standard groundwater samples would be an integral part of the sampling and analysis program.

12.4.4. Air Sparging

This technology involves the injection of air under pressure below the water table using drilled or pushed wells. The goal is to provide adequate air distribution to allow the volatilization of VOCs from groundwater and, in some cases, the dissolution of oxygen into the groundwater. Vapors are often collected with a soil vapor extraction system.

The analytical requirements would include the monitoring of DO and oxidation-reduction potential in groundwater as an indirect indication of nearby air-flow paths. Such measurements are best made downhole using suitable probes or low-flow sampling techniques. For some projects, tracers such as helium or sulfur hexafluoride may be added to the injected air and monitored for in vapors or possibly in groundwater samples. Monitoring of target contaminant in standard groundwater samples would be an integral part of the sampling and analysis program.

12.4.5. In situ Thermal Remediation

In situ thermal remediation is an aggressive soil and groundwater treatment technology that involves heating of the subsurface. The heating can be achieved through:

- The introduction of electric currents with resultant resistive heating of the soil
- The injection of steam, or
- The placement of high temperature electric conductive heaters into the subsurface.

The heating raises the vapor pressure of contaminants, speeds diffusion, reduces viscosity and possibly interfacial tension for non-aqueous phase liquids, and possibly increases contaminant solubility. All of these effects increase the rate of contaminant mass removal from the subsurface. The remediation methods use vapor extraction and, in the case of steam injection, liquid extraction to remove mass from the subsurface. Steam and vapors are typically condensed as part of the above-ground treatment. The analytical program includes the monitoring of contaminant concentrations in both the collected vapors and groundwater (if heating is conducted below the water table). Sampling, analysis, and data collection would include the measurement of contaminant concentrations, collection of temperature data (usually from subsurface thermocouples), and sampling from above-ground treatment equipment, including analysis of condensed liquids and non-condensable gases.

The analytical program must be prepared for handling of high contaminant concentrations in the media of interest as mass removal rates are often very high. The sampling program, both for groundwater monitoring and process monitoring, must deal with sampling of media at high temperatures. To assure safe sampling and to avoid excessive losses of contaminants, sampled groundwater must be cooled before transfer to sample containers. Refer to USACE Engineer Manual EM 1110-1-4015 for additional discussion of sampling approaches.

12.5. Ex situ Soil Treatment

Rapid treatment processes such as solidification/stabilization (S/S) and low-temperature thermal desorption, rely primarily on pre- and post-treatment sampling and analysis of soil to confirm treatment criteria have been achieved. Relatively slow treatment processes, such as landfarming, usually require periodic monitoring to assess the progress of treatment over long periods. For landfarming and thermal desorption, post-treatment sampling must demonstrate that an adequate degree of contaminant reduction has occurred. S/S does not entail contaminant destruction, but sampling and analysis is usually still required to demonstrate that the leachability of contaminants has been adequately reduced.

12.5.1. Off-Site Disposal

Prior to off-site disposal of contaminated soil, sampling and analysis is usually required to demonstrate that the contaminated soil meets acceptance criteria for the landfill or other TSDF. Sampling and analysis may also be necessary to comply with transportation regulations. After excavation, the soil is typically placed in a covered stockpile, or in a roll-off container, while it is being staged for off-site transportation. Sampling is typically performed using hand tools (e.g., trowel, hand auger, etc.), and the soil samples are usually sent to an off-site laboratory. Incremental sampling of the soil as it is placed in the container or stockpile will yield more representative concentrations.

12.5.2. Landfarming

Landfarming (sometimes referred to as land treatment) typically involves placement of contaminated soil onto a lined cell where it is periodically irrigated and tilled to promote biodegradation of organic contaminants. Landfarming is a relatively slow treatment process that requires periodic monitoring to assess the progress of treatment. The length of the treatment period depends on what types of contaminants are present, concentrations of contaminants, and on the local climate. Treatment periods can range from a few months to two or more years. In cold climates, treatment operations are usually suspended during the winter and not resumed until spring. The contaminants of concern represent the primary monitoring parameters. In some instances, it may be possible to correlate contaminant levels to a surrogate parameter such as total petroleum hydrocarbons (TPH). There are commercially available field analysis kits for parameters such as TPH, and PAHs. The field analysis kits can often be used to monitor the progress of treatment, and to determine when to collect confirmatory soil samples for laboratory analysis. Other important monitoring parameters include: soil moisture content, soil pH, and soil nutrient levels (e.g., nitrogen and phosphorous). Sampling is typically performed using hand tools (e.g., trowel), and the confirmatory soil samples are usually sent to an off-site laboratory. For more information, refer to the following:

- UFGS 02 54 20 (02190), Bioremediation of Soils Using Landfarming Systems.

12.5.3. Solidification/Stabilization

Solidification/Stabilization (S/S) typically involves blending contaminated soil with portland cement and/or other additives, in order to reduce the leachability of metals. A pug-mill is often used to bend the soil and the cement. S/S does not entail contaminant destruction, but sampling and analysis is required to demonstrate that the leachability of contaminants has been adequately reduced. The contaminants of concern (usually metals) represent the primary monitoring parameters. Sampling can usually be performed using hand tools, and the soil samples are usually sent to an off-site laboratory.

For more information, please refer to:

- UFGS 02 55 00 (02160), Solidification/Stabilization (S/S) of Contaminated Material (04-2006); and ETL 1110-1-158, *Treatability Studies for Solidification/Stabilization of Contaminated Material.*

12.5.4. Low-Temperature Thermal Desorption

Low-temperature thermal desorption (LTTD) is a process that involves heating soil to rapidly achieve stringent cleanup criteria for organic contaminants. During the heating process, the contaminants are volatilized from the soil, and then condensed out and recovered for off-site disposal. A limited degree of contaminant destruction may occur for some of the more thermally labile contaminants, which may have escaped as heated vapor. LTTD systems include complex cooling and vapor treatment systems. The heated vapor that is drawn off of the soil must be cooled, has to pass through particulate filtration systems, and usually through vapor-phase GAC adsorption vessels.

During operation of LTTD units, continuous emissions monitoring (CEM) is usually required. Stack gas CEM monitoring parameters usually include: oxygen, carbon monoxide, total hydrocarbons, and nitrogen oxides. Testing for particulate matter (e.g., PM_{10}) in the stack gas is also usually required. During the proof of performance (POP) test, testing of the stack gas for contaminants of concern is usually required.

The contaminants of concern represent the primary monitoring parameters. Soil samples are usually collected at a designated point of the conveyor system for feeding the soil into the LTTD unit, and from the treated soil stockpile. Sampling can usually be performed using hand tools, and the soil samples are usually sent to an off-site laboratory. For more information, please refer to:

- ETL 1110-1-173, *Thermal Desorption*, 31 May 1996.

12.6. In situ Soil Treatment

12.6.1. Soil Vapor Extraction (SVE)

Soil vapor extraction is a technology used to remediate soils above the water table and involves the application of a vacuum to wells screened above the water table and extraction of VOC vapors. Monitoring largely consists of the sampling and analysis of soil gas concentrations and measurement of vacuum distribution using sensitive vacuum gauges (down to a few millimeters of water column). Soil gas sampling is conducted from monitoring probes, from individual extraction wells, and from combined vapor flow at the above-ground treatment plant. Sampling of soil gas from subsurface probes can be conducted using Summa™ canisters, vacuum boxes, and Tedlar™ bags, or through direct-reading instruments (e.g., a PID) that can create vacuums greater than observed in the probes. Sampling of vapors being withdrawn from individual extraction wells or combined influent at the treatment facility usually involves the use of a vacuum box, sample pump, or Summa™ canisters, as the applied vacuums may be more than a half atmosphere. Any sampling pump used to collect the samples and transfer vapors to a container such as a Tedlar™ bag must not introduce potential contaminants such as oils.

12.6.2. Bioventing

Bioventing generally involves injection of atmospheric air into the soil above the water table to promote aerobic biodegradation of contaminants (where ambient conditions are oxygen limited), particularly petroleum hydrocarbons. The injection of gases other than, or in addition to atmospheric air can promote aerobic cometabolic degradation or anaerobic degradation of some chlorinated hydrocarbons. Tracer gases such as helium or sulfur hexafluoride are occasionally injected to assess the flow paths and diffusive and dispersive losses. Sampling in support of bioventing design and operation normally involves the collection of soil gas samples. Active soil gas sampling would be conducted in sampling ports at different locations and depths to assess distribution of oxygen, contaminant vapors, or other injected gases. Carbon dioxide, methane, or other gases may also be of interest to assess the degradation processes. Analysis for oxygen, carbon dioxide, and methane would typically be conducted in the field using multi-gas testing instruments.

Contaminant vapor concentrations are also determined to assess progress and the potential displacement of hazardous vapors to possible exposure points. Contaminant vapors may be analyzed in the field using non-specific analytical equipment (e.g., PID) or samples may be taken in Tedlar™ bags or Summa™ canisters for fixed or mobile lab analysis. Soil sampling for fixed or mobile lab analysis may be necessary to determine attainment of treatment goals.

12.6.3. In situ Thermal Remediation.

The requirements are similar to those for in situ thermal remediation for groundwater. Please refer to Section 12.4.5.

12.7. Chemical and Physical Parameters for Remediation Processes

Early site characterization up through and including the Remedial Investigation (RI) or RCRA facility Investigation (RFI) often focuses on obtaining data strictly relating to the contamination at a site. These investigations concentrate on gathering information such as concentration, extent of the contamination, potential sources, exposure scenarios, etc. Physical and chemical data of the medium relevant to remedy implementation are often overlooked. For example, if air stripping or carbon adsorption is being considered for removal of VOCs from the groundwater, it is important to know the concentration of iron and calcium in the groundwater, because these chemicals can cause fouling and plugging problems, which increase the cost of operations and maintenance (O&M). Site history and other information gathered during early phases of site characterization should provide ideas as to the contamination and the possible remedial processes that will be considered. Generally, cost savings can be achieved by collecting process characterization samples during initial field sampling events.

The number of process samples taken for analysis during the initial field sampling events should be kept to a minimum to save time and costs. Parameters that can be used as estimates for other parameters should be used. For example, the humic content of the soil or the COD may be required for the final design of a selected process. However, TOC is often recommended in the earlier stage of investigation instead of these parameters, as it is fairly easy to obtain and should provide an estimate of these parameters for the initial data analysis. For metals, consider having a laboratory to analyze for all the metals (approximately 30), as it may not cost any more than analyzing for a selected few. Analyzing for some parameters in the field may provide immediate information as to additional samples to take to eliminate the need for an additional trip to the field. It is recommended that the following water parameters be routinely analyzed during an early field sampling event: temperature, pH, conductivity, turbidity, DO, and oxidation-reduction potentials. These parameters can provide much information on the water and are easily measured with probes or other simple field instruments. It is also recommended that soil samples be collected for sieve analysis during the initial sampling event. Sieve analysis can provide a rough estimate of the following parameters: plasticity, field capacity, bulk density, particle density, permeability, porosity, and soil classification.

ASTM D 7294 lists physical and chemical treatment processes design data needed to evaluate, select, and design treatment processes for remediation at contaminated sites. It is recommended that these data are collected prior to evaluating remedies for large return/savings by investing very little time and money up front. This additional information will allow the decision makers to screen out technologies that are not applicable (e.g., SVE in tight soil) or cost effective, determine more accurate cost estimates, and save time and money by not having to re-mobilize to collect data during the design phase.

The analytical costs and time of obtaining these samples will be less than that for obtaining contaminant samples, because screening-level quality data is often sufficient, many tests are simple and can be performed in the field, and a minimal number of samples are needed to provide the design data required. Additionally, if off-site process parameters are expected to be the same as on-site parameters, samples can be taken from uncontaminated areas of the site and do not need to be handled as hazardous waste.

ASTM D 7294 identifies the most important parameters for a particular media and technology. Use the tables in ASTM D 7294 as a guide in conjunction with the Treatment Technology Profiles in the Remediation Technologies Screening Matrix and Reference Guide at http://www.frtr.gov/matrix2/section1/toc.html to quickly search and screen technologies that may be evaluated for site remediation later. The process data needed to further evaluate these technologies can then be obtained from ASTM D 7294 and can then be easily collected in the field at the same time as the contaminant data collection.

The laboratory methods listed in ASTM D 7294 are well defined and consist primarily of Standard Methods, SW-846, and EPA 600 methods. The field methods are not as well defined, and the table gives general information on field methods and their time and cost. Manufacturer’s literature, the Internet and other information should be used to select the field kits that best meet the analytical and cost needs for that site.

For further information, see:

- ASTM D 5463 (Standard Guide for Use of Test Kits to Measure Inorganic Constituents in Water).

12.8. References

- Technical and Regulatory Guidance for In Situ Chemical Oxidation of Contaminated Soil and Groundwater, Second Edition (January 2005).
- DG 1110-1-2, Adsorption Design Guide, 1 March 2001.
- DG 1110-1-3, Engineering and Design-Air Stripping, 31 October 2001.

- ETL 1110-1-158, Treatability Studies for Solidification/Stabilization of Contaminated Material, 28 February 1995. http://www.cement.org/waste/pdfs/USACETreatabilityGuide.pdf
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- ETL 1110-1-173, Thermal Desorption, 31 May 1996.
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- Multi-Phase Extraction: USACE Multi-Phase Extraction Engineer Manual EM 1110-1-4010, 1999.
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Chapter 13. Field Testing

13.1. Purpose

This chapter provides procedures for performing field testing to accurately characterize site conditions.

13.2. Scope

The scope of this chapter includes a discussion of field screening and field testing, and gives examples of SOPs for several analyses that are required by various regulations to be performed in the field. Other analyses that are not required for regulatory compliance also may be performed in the field. Field testing procedures and strategies should be specified in the project SAP.

13.3. Background

A sampling event is conducted to collect sample material for analysis of specific parameters to determine site conditions. Some of these parameters can influence the characteristics of the contaminants that need to be investigated and, therefore, must be accurately measured at the time of the sampling event. Certain parameters require analysis in the field because of the potential for sample deterioration during transport to a laboratory. Some parameters must be analyzed in the field because they can fluctuate rapidly with time and sample conditions. Parameters that may require measurement in the field include pH, temperature, residual chlorine, specific conductance, oxidizer/cyanide test, and DO.

13.4. Training

Personnel who will be performing any field testing must be trained in sample collection, preparation, and field testing methods, and demonstrate proficiency in the SOP for the test to be completed. Training and proficiency must be documented in accordance with the SAP. Navy personnel should also document their training following the Uniform Standards for Sampling, OPNAVINST 5090.1C, Environmental and Natural Resources Program Manual, Chapter 29.

13.5. Field Screening

Field screening procedures are typically qualitative or semi-quantitative in nature and are performed using special screening equipment or techniques, such as probes or portable hand-held instruments and meters. Some field screening is performed using field testing methods, and special kits that are designed for use in a field environment. Field screening is used to provide a quick determination of the presence, magnitude, or absence of an analyte of concern. Field screening provides preliminary results with minimized costs on a real-time or near-real-time basis. Because the quality control and analytical sophistication of field screening is not as controlled as it is for laboratory testing, a representative set of split or duplicate samples should be submitted to a laboratory for comparison of the results.

Examples of field screening SOPs are located in Section 13.7 and are summarized in Table 13-1.

Table 13-1. Field Screening Methods

Field Test	Matrix	Test Event
Specific Conductance	Water	CERCLA and drinking water assessment
Total Residual Chlorine	Water	Drinking water and wastewater assessment
pH	Water, aqueous wastes	Waste profiling and wastewater assessment
Oxidizer/ Cyanide	Wastewater, aqueous wastes	Waste profiling and wastewater assessment
Temperature	Water	All water sampling events
PID	Water, soil, waste	Preliminary screening for unknown volatile organic contaminants

There is a wide variety of field screening tests available, and the list is growing, but the analyst must be very cautious and clearly define and control the use of any data generated. The chance for false positives and false negatives must be known, and adequate quality control must be demonstrated to ensure approval of the screening procedures for use by the regulatory authority. Positive and negative measurements usually are acceptable as long as the Limit of Detection (see DoD Quality Systems Manual) associated with non-detects is below the action level or regulatory threshold limit. The best results for field screening methods are obtained when the contaminant is known and the technique is acceptable for use under the environmental conditions at the site.

13.6. Field Operations

Field operations that include field screening or field testing should consider the following:

- QAPP
- Test methods
- Calibration procedures
- Reference standards
- Materials and material preparation
- Sample storage
- Sample handling
- Sample traceability
- QC samples
- Control charts
- Data quality criteria
- Detection limits
- Recordkeeping
- Data reporting procedures
- Environmental conditions
- Facility conditions and control
- Personnel qualifications
- Disposal
- Safety.

13.7. Sample SOPs

The remainder of this chapter provides sample SOPs for the following.

- **Figure 13-1:** Specific Conductance (Field Testing)
- **Figure 13-2:** Field Determination of Total Residual Chlorine
- **Figure 13-3:** Field pH Measurements Using pH Paper
- **Figure 13-4:** Field pH of Aqueous Samples by Electrometric Measurement
- **Figure 13-5:** Oxidizer Field Testing for Cyanide Samples
- **Figure 13-6:** Field Temperature Determination
- **Figure 13-7:** Calibration & Maintenance of a Photovac PID.

These SOPs are written for specific instruments. If using a different instrument, the procedure may differ. Consult the Instrument Manual, and any appropriate regulatory guidance.

Figure 13-1. Sample SOP for Specific Conductance (Field Testing)

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Specific Conductance
(Field Testing)

Prep By:
Approved By:
Effective Date:

CAUTION

This Standard Operating Procedure has been prepared as an example for this handbook and may not be specifically applicable to all activities or organizations. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Figure 13-1. Sample SOP for Specific Conductance (Field Testing) (continued)

1.0 Title: Standard Operating Procedure for Performing Specific Conductance Testing (Field Testing).

2.0 Scope and Application

2.1 This procedure is applicable to drinking, ground, surface, and saline waters; and domestic and industrial waste. This procedure is for field testing where needed; however, some regulations do allow this test to be performed in the laboratory. RCRA and CERCLA require analyses to be performed immediately upon sample collection. Drinking water programs allow a 28-day holding time until analysis. See these regulations for sample preservation requirements.

3.0 Summary of Method

3.1 The determination of specific conductance gives a measure of the ability of an aqueous solution to carry an electric current, which is related to the ions present in the aqueous solution.

3.2 The specific conductance of a sample is measured using a self-contained conductivity meter (Wheatstone Bridge-type or equivalent).

3.3 Whenever possible, samples are analyzed at 25°C. If samples are analyzed at different temperatures, corrections must be made and results reported at 25°C.

4.0 Interferences

4.1 Platinum electrodes can degrade and cause erratic results. When this happens, as evidenced by erratic results or flaking of the platinum black, the electrode should be replaced.

4.2 The specific conductance cell can become coated with oil and other materials. It is essential that the cell be thoroughly rinsed and cleaned between samples.

5.0 Equipment List

5.1 Conductivity Bridge (self-contained, Wheatstone Bridge-type or equivalent. Range 1–1,000 µS/cm) with Conductivity Cell (cell constant of 1.0, YSI #3403 or equivalent). Current METCAL calibration is required. Annual verification of cell constant is required.

5.2 Sample container (glass or plastic) able to submerge cell probe completely.

5.3 Thermometer - calibrated and able to read ± 0.1 degrees. (At a minimum, mercury in glass thermometers and dial-type thermometers shall be checked annually against a National Institute of Standards and Technology (NIST)-traceable thermometer.)

5.4 FLB/FN

5.5 Conductivity meter calibration log book

5.6 Sample collection device (e.g., bailer, scoop).

6.0 Reagents and Materials

6.1 Potassium chloride (KCL) solution; 0.01M: Dissolve 0.7456 gram of predried (2 hours at 105°C) KCL (reagent grade) in distilled water, and dilute to 1 liter at 25°C. Conductivity of this solution is 1,413 µS/cm at 25°C. Laboratory personnel can prepare this solution, or an equivalent NIST-traceable reference standard may be purchased.

6.2 Distilled water. If doing RCRA Method 9050, deionized distilled water is required with conductivity of less than 1 µmho/cm.

Figure 13-1. Sample SOP for Specific Conductance (Field Testing) (continued)

7.0 Hazards and Safety Precautions

7.1 Refer to the MSDS for details on chemical hazards and personal protection.

7.2 Minimum PPE, (i.e., chemical safety goggles and appropriately selected protective gloves) should be worn. Other PPE (e.g., safety shoes, hard hat, respirator) will be dictated by the specific hazards of the sampling site.

7.3 Emergency eyewash/shower must be present, as appropriate.

8.0 Preparation

8.1 Turn instrument on, and allow to warm up.

8.2 Ensure conductivity cell is correctly attached to conductivity meter.

8.3 Follow the directions of the manufacturer for the operation of the instrument.

9.0 Calibration and Standardization

Note: This procedure to be performed only by properly trained sampling personnel.

9.1 Prior to sampling, conduct the following:

9.1.1 Verify conductivity meter has current METCAL calibration.

9.1.2 Calibrate conductivity meter prior to use, using KCl solution (see paragraph 6.1) at 25°C and the table below to check the accuracy of the cell constant and conductivity bridge:

Conductivity 0.01M KCl	
°C	**µS/cm**
21	1305
22	1332
23	1359
24	1386
25	1413
26	1441
27	1468
28	1496

9.1.3 Enter the following calibration data in meter log book:

- Meter reading
- Temperature
- Reference material or calibration standard source
- True value/temperature of the calibration standard expected
- Signature and date
- Verified within manufacturer’s tolerances

9.1.4 The number and concentration of the standards used for calibration should be based on the testing needs for the field activity. A single point check standard in the range of the sample concentrations to be measured may be used for field screening.

Figure 13-1. Sample SOP for Specific Conductance (Field Testing) (continued)

10.0 Procedure

10.1 Obtain sample according to the appropriate sampling SOP, and pour into sample container with a minimum of aeration.

10.2 Rinse cell with distilled water. Rinse probe with remaining sample water.

10.3 Place specific conductance cell in sample container.

10.4 Ensure sample level is above cell air vent hole.

10.5 Ensure all air is vented from cell.

10.6 Follow manufacturer's directions for operation of the conductivity meter.

10.7 Determine the temperature of samples. If the temperature of the samples is not 25°C, make temperature correction in accordance with the following guidelines:

- If temperature of the sample is below 25°C, add 2% of the reading per degree.
- If temperature is above 25°C, subtract 2% of the reading per degree.

The more the sample temperature of the measurement deviates from 25°C, the greater the uncertainty in applying the temperature correction.

10.8 Record results in the FLB/FN

- Sample ID
- Sample temperature, °C
- Meter reading, µmhos/cm
- Corrected meter reading, µmhos/cm at 25°C
- Signature and date
- SOP reference name/number.

10.9 Recheck calibration with the KCl solution (see paragraph 6.1), and enter into conductivity meter log book.

11.0 QA/QC

11.1 Conductivity cells must be kept clean.

11.2 Conductivity cell constant must be determined annually per Reference 12.2, Section 7.1.

11.3 Temperature variations and corrections represent the largest source of potential error.

11.4 Analyze calibration standards (see paragraph 6.1) after approximately every 15 samples, per RCRA method #9050A (paragraph 8.2).

11.5 Run one duplicate sample for every 10 samples, per RCRA method #9050A (paragraph 8.3).

12.0 References

12.1 EPA—Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020 Method 120.1 (Specific Conductance µmhos at 25°C) Editorial Rev., 1982. Applicable to drinking water and wastewater.

12.2 Test Methods for Evaluating Solid Wastes. SW-846, Method 9050A Specific Conductance. Applicable.

Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Field Determination of Total Residual Chlorine

Prep By:
Approved By:
Effective Date:

CAUTION

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Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine (continued)

1.0 Title: Standard Operating Procedure for Field Determination of Total Residual Chlorine.

2.0 Scope and Application

2.1 The HACH DR 100 Colorimeter is a portable battery-powered colorimeter designed for field testing. This instrument is utilized to measure the total residual chlorine content of samples. The DR100 conforms to the requirements of reference 11.1 using a 2.5-cm sample cell and DPD pillow-packed reagents for total residual chlorine determination. Reference 11.2 details operation and maintenance for the DR100.

2.2 This procedure is used to determine the level of residual chlorine for drinking and wastewater samples. Residual chlorine in drinking water must meet specified requirements. In wastewater analysis, residual chlorine can possibly interfere with some test procedures.

3.0 Summary of Method

3.1 Chlorine (hypochlorite ion, hypochlorous acid) and chloramines stoichiometrically liberate iodine from potassium iodide at pH 4 or less. The liberated iodine reacts with N,N-diethyl-p-phenylene diamine (DPH) to produce a red-colored solution. The solution is spectrophotometrically compared to a series of standards. The results are read in mg/l Cl.

4.0 Interferences

4.1 Interferences from oxidized manganese and copper are corrected by the addition of the DPD reagents. The method inhibits trace metal catalysts. High concentrations of monochloroamine (combined chlorine) will not interfere with the total residual chlorine test.

4.2 Turbidity and color will interfere with the colorimetric analysis.

5.0 Equipment

5.1 DR100 HACH Colorimeter with calibration curve.

5.2 Clippers.

5.3 2.5-cm sample cell.

5.4 1.0-cm cell holder.

5.5 1.0-cm sample cell.

5.6 Stopwatch or wrist watch.

5.7 Various appropriate pipettes.

5.8 100-mL volumetric flasks.

5.9 FLB/FN

6.0 Reagents and Materials

6.1 All reagents must be reagent-grade chemicals unless otherwise specified.

6.2 Stock Potassium Permanganate Solution (1000 ppm Equivalent–Chlorine Standard Solution). Place 0.891 gram $KMNO_4$ in a 1-liter volumetric flask, and dilute to 1 liter. Consult Reference 11.1 for preparation. This solution must be refrigerated and has a shelf life of 6 months. This reagent can be prepared by laboratory personnel or purchased from commercial sources.

6.3 DPD total chlorine reagent powder pillows. (Purchased from HACH)

Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine (continued)

6.4 Sodium Hydroxide, 0.1N. This reagent can be prepared by laboratory personnel or purchased from commercial sources.

6.5 Nitric Acid, 0.1N. This reagent can be prepared by laboratory personnel or purchased from commercial sources.

6.6 Reagent (deionized) water.

7.0 Hazards and Safety Precautions

7.1 Refer to the MSDS for details on chemical hazards and personal protection.

7.2 Minimum PPE is chemical safety glasses and appropriately selected gloves. Hazards associated with the sample or sampling site may require additional PPE.

7.3 An emergency eyewash/shower should be present, as appropriate.

8.0 Calibration and Standardization

8.1 Standard Preparation

8.1.1 Pipet 10.0 mL of the primary stock standardization solution potassium permanganate (paragraph 6.2) into a 100-mL volumetric flask. Fill volumetric flask to mark with reagent (ASTM Type I or II) water and mix. This is the secondary stock solution (equivalent 100 ppm chlorine).

8.1.2 Pipet 1.5 mL of secondary stock solution (paragraph 8.1.1) into a 100-mL volumetric flask. Fill flask to volume with reagent (ASTM Type I or II) water, and mix. This will produce an equivalent total chlorine standard with a concentration of 1.50 mg/L.

8.1.3 Pipet 1.0 mL of secondary stock solution (paragraph 8.1.1) into a 100-mL volumetric flask. Fill the flask to volume with reagent (ASTM Type I or II) water, and mix. This will produce an equivalent total chlorine standard with a concentration of 1.00 mg/L.

8.1.4 Pipet 50.0 mL of the 1.00 mg/L standard (paragraph 8.1.3) into a 100-mL volumetric flask. Fill the flask to volume with reagent (ASTM Type I or II) water, and mix. This will produce a total chlorine standard with a concentration of 0.50 mg/L.

8.1.5 Pipet 10.0 mL of the 1.00 mg/L chlorine standard (paragraph 8.1.3) into a 100-mL volumetric flask. Fill the flask to volume with reagent (ASTM Type I or II) water, and mix. This will produce a total chlorine standard with a concentration of 0.10 mg/L.

8.2 DR100 Calibration. This is completed by laboratory personnel.

8.2.1 Using the DR100 and the procedure in paragraph 8.3.3, STEPS (1) through (10), analyze a blank (0.00 mg/L ASTM Type I or II water) and the 0.10, 0.50, 1.00, and 1.50 mg/L standards. (8.1.2, 8.1.3, 8.1.4, 8.1.5).

8.2.2 After reading each standard, place the cell contents used for each reading in a flask, and titrate with standardized ferrous ammonium sulfate (FAS) titrant. Record these values as the true standard concentrations.

8.2.3 Plot a calibration curve of measured (meter) values versus true values. This chart shall be kept with the meter and updated annually, or when quality control data indicates potential errors.

8.3 Daily Standardization

Note: Prior to beginning this procedure, ensure reagents are not beyond expiration date.

8.3.1 Once the meter has been checked to ensure that it is in acceptable working condition, it is ready for daily standardization. The DR100 Colorimeter does not require a warm-up period. Both the mechanical zero and low battery indicator check should be made with the meter in the same general position that it will be in when the measurements are made. (See paragraph 10.3.3).

8.3.2 The DR100 Colorimeter shall be standardized prior to use each day at a minimum of 3 points that bracket the

Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine (continued)

expected sample value range. Normally, use the following standard concentrations: 0.00 mg/L (ASTM Type I or II water), 0.50 mg/L, and 1.00 mg/L. This standardization procedure shall be performed before the meter can be used in making field measurements. Entries are to be made in the FLB/FN indicating the standards used, the respective meter readings, and the corrected values. The corrected value is the value obtained from the calibration chart that corresponds to the meter reading. Each meter shall have an accompanying calibration chart for that meter.

8.3.3 Standardization Steps

STEP (1) Open the light shield, turn the right set knob fully clockwise, and place the 1-cm cell holder (without the 1-cm cell in the holder) in the left set position of the sample well. Close the light shield.

STEP (2) Hold the ON button down while adjusting the left set knob to align the meter needle with the arrow at the extreme left of the scale arc. Remove the cell holder.

STEP (3) Fill a clean 2.5-cm sample cell with reagent (ASTM Type I or II) water. Cap the cell and place it into the sample well. Press down firmly to seat the sample cell, and close the cell holder.

STEP (4) Hold the ON button down while adjusting the right set knob for a reading of 0.00 mg/L. Open the light shield, and remove the sample.

STEP (5) Remove the cap from the sample cell used in STEP (3) and add the contents of a DPD total chlorine powder pillow to the sample cell. Cap and shake it for 20 seconds. Allow at least 3 minutes but not more than 6 minutes for proper color development. (This is a “prepared standard” of 0.00 mg/L or “blank”.)

Note: It is not necessary for all of the powder reagent particles to dissolve to obtain an accurate reading. Shaking the cell dissipates bubbles that may form on the cell wall and interfere with the test results.

STEP (6) Place the cell containing the prepared standard from STEP (5) into the sample well. Press down firmly to seat the sample cell, and close the light shield.

STEP (7) Hold the ON button down until the meter stabilizes.

STEP (8) Read the total chlorine concentration in mg/L from the upper (0.00–2.00) mg/L) scale. If the meter reading is 0.05 mg/L or less, then the meter is within the acceptable range and is to be reported as 0.00 mg/L.

STEP (9) Record this as a meter reading in the FLB/FN under the blank total chlorine concentration standard.

STEP (10) Remove the sample cell from the sample well, and rinse the inside of the sample cell and cap with reagent (ASTM Type I or II) water at least two times to remove any residue.

8.3.4 Repeat STEPS (3) through (10) using the 0.50 mg/L and the 1.00 mg/L standards (prepared in 8.1) in place of the ASTM Type I or II water. Record each meter reading in the FLB/FN. Refer to the calibration table to determine the true values associated with the meter readings, and record in the FLB/FN. Corrected values must be within the acceptable range of +0.05 units of the known standard concentration.

8.3.5 If the corrected value is outside of the acceptable range of +0.05 mg/L from the known standard concentration, then another standard solution shall be prepared and the standardization repeated. If the corrected value remains outside of this range, then it shall be assumed that the meter is malfunctioning. The Lab Director should be contacted for further instructions.

9.0 Procedure

9.1 Sample Collection, Preservation and Handling

9.1.1 Samples shall be collected in a clean glass or plastic container and tested on site within 15 minutes. No preservation is necessary for on-site testing

9.1.2 Samples should have a pH between 6 and 7 for this procedure to be used. If necessary, an appropriate amount of acid (0.1 N Nitric Acid) or base (0.1 N Sodium Hydroxide) that does not contain ammonium or chloride ions can be used to

Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine (continued)

adjust the pH to within this range.

9.1.3 Calibration curves shall be established for each meter (see paragraph 8.2) prior to initial use. Calibration curves should be updated annually.

9.1.4 Prior to daily use, standardize meter using standardization procedure (see paragraph 8.3).

9.2 Sample Analysis

9.2.1 Perform STEPS (3) through (10) of paragraph 8.3.3 for each set of samples and if the meter is physically relocated or disturbed during analysis of a sample set (any movement may offset the prism and invalidate the analyses). Analyze each sample following STEPS (1) through (10) of paragraph 8.3.3.

9.2.2 Once the meter value for each sample has been determined, that value shall be found in the calibration curve/table accompanying each meter. This gives the true total chlorine concentration. Record this true concentration in FLB/FN or appropriate data sheet.

9.2.3 If the sample temporarily turns yellow when adding the DPD Total Chlorine Reagent or reads above the highest scale division, the chlorine concentration is too high for the meter calibration. Any readings above 2.00 mg/L should be reported as >2.00 mg/L. Chlorine concentrations may be approximated by additional sampling, dilution, and further analysis. The sample also may be retained to determine if an interference exists or for reanalysis using a different test method.

10.0 Quality Assurance for Hach DR100

10.1 Ten percent of the samples shall be analyzed in duplicate (split samples). Ten percent of the samples shall have duplicate samples taken and analyzed (field duplicates). Record data in the FLB/FN or appropriate Data Sheet. Follow QAPP for reporting results.

10.2 A calibration curve is prepared at least annually. When any meter measurement is made, the value from the meter should be read to the nearest 0.05 units up to a meter reading of 1.50 mg/L. All values between 1.5 and 2.0 mg/L should be read to the nearest 0.1 unit on the meter scale. The meter value obtained in this procedure should then be found on the accompanying calibration curve to determine the true total residual chlorine concentration.

10.3 The HACH DR100 Colorimeter should have routine maintenance performed before it is standardized or used for field measurements. The following maintenance steps cover the internal components to ensure that the meter is functioning properly.

10.3.1 The DR100 Colorimeter uses four “AA” size batteries. These batteries should be replaced at least annually. The meter should be tagged to indicate the date of the last battery change. Do not use the instrument when the “Low Battery” indicator is lit. An indicator light that remains lit after batteries have been replaced may indicate that the internal lamp is burned out. In any event, if the light remains on after batteries have been changed, the meter is not functioning correctly and should not be used. The Lab Director should be contacted immediately.

10.3.2 While the meter is in the OFF position and as level as possible, the meter needle should align with the arrow at the far left of the meter scale. To reset the meter needle, use a screwdriver to adjust the mechanical zero screw in the center of the meter.

10.3.3 Other important considerations in obtaining accurate data are the condition of the sample cell holder and the sample cells. The exterior of the sample cell holder and sample cells should be cleaned with an absorbent tissue before placing them into the meter. Both the sample cell holder and the sample cell should be kept as clean and free of scratches as possible. After each test, the sample cells must be rinsed thoroughly with ASTM Type I or II water and dried. At no time should the sample cell be left in the meter after completion of a test. As with any analog output meter, the meter should be directly in front of the operator and as level as possible to eliminate errors when reading the meter scale.

11.0 References

11.1 Standard Methods for the Examination of Water and Wastewater, 21st Edition; Method 4500-C1.G, DPD Colorimetric Method.

Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine (continued)

11.2 DR100 HACH Colorimeter Total Chlorine Test Kit Instruction Manual.

Figure 13-2. Sample SOP for Field Determination of Total Residual Chlorine (continued)

Data Sheet
Total Residual Chlorine

Total Residual Chlorine DR100 S/N_____________

Daily Standardization

Date:_______________	*Meter Reading mg/L*	*Corrected Value, mg/L*
Blank Total Chlorine Conc.	______________________	___________________________
1.00 mg/L Total Chlorine Std.	______________________	___________________________
0.50 mg/L Total Chlorine Std.	______________________	___________________________

Sample Analysis

Sample Description	*Meter (mg/L)*	*Corrected (mg/L)*	*Initials/Date/Time*

Figure 13-3. Sample SOP for Field pH Measurements Using pH Paper

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Field pH Measurements Using pH Paper

Prep By:
Approved By:
Effective Date:

CAUTION

This Standard Operating Procedure has been prepared as an example for this handbook and may not be specifically applicable to all activities or organizations. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Figure 13-3. Sample SOP for Field pH Measurements Using pH Paper (continued)

1.0 Title: Standard Operating Procedure for pH Measurements Using pH Paper.

2.0 Scope and Application

2.1 The pH paper method is used as a pH-screening method for aqueous wastes or in cases where pH measurements using a meter are not possible.

2.2 This method is not applicable to wastes that contain components that may mask or alter the pH paper color change.

2.3 pH paper is not considered to be as accurate in measuring pH as a pH meter. Measurements taken using paper can be used to define waste as corrosive or non-corrosive (pH < 2 or > 12.5) only if the measured values differ from either threshold limit by a full pH unit. If readings within the ranges of either pH 1–3 or pH 11.5–13.5 are obtained using pH paper, then a pH meter should be used if possible to determine whether the waste is corrosive.

3.0 Summary of Method

3.1 The approximate pH of the waste is determined with wide-range pH paper.

3.2 A more accurate pH determination is made using “narrow-range” pH paper whose accuracy has been determined by using a series of buffers or by comparison with a calibrated pH meter.

4.0 Interferences

4.1 Certain waste may inhibit or mask changes in the pH paper. This interference can be determined by adding small amounts of acid or base to a small aliquot of the waste and observing whether the pH paper undergoes the appropriate changes.

4.2 Identification of Interferences

CAUTION: Addition of acid or base to samples may result in violent reactions or the generation of toxic fumes. Extreme caution must be exercised. Do not perform this test when cyanides (or other toxic fumes) may be present or generated. Perform this test in the field only with appropriate protective equipment and adequate ventilation.

4.2.1 Collect two samples of approximately 2 mL each.

4.2.2 Place pH paper (wide or narrow range) in each.

4.2.3 Add acid drop wise to the first sample until a pH change is observed by noting the color change.

4.2.4 Add base drop wise to the second sample until a pH change is observed by noting the color change.

4.2.5 The observation of the appropriate color change is a strong indication that no interferences have occurred. Record the results in the FLB/FN.

5.0 Equipment

5.1 PPE (see paragraph 7).

5.2 A sample collection device, if necessary.

5.3 Sample containers, if necessary.

5.4 FLB/FN.

6.0 Reagents and Materials

6.1 Wide range pH paper.

6.2 Narrow range pH paper (every 0.5 pH unit).

Figure 13-3. Sample SOP for Field pH Measurements Using pH Paper (continued)

6.3 Certified pH buffers.

6.4 Dilute acid (e.g., 1:4 HCl) for identification of interferences.

6.5 Dilute base (e.g., 0.1N NaOH) for identification of interferences.

6.6 Drum opening tools, if necessary.

7.0 Health and Safety Precautions

7.1 Corrosives damage skin/tissue and should not be touched or inhaled.

7.2 For known or suspected corrosives where pH meter readings are not possible, wear the following PPE:

7.2.1 Full body protection (Tyvek®).

7.2.2 Impervious rubber gloves.

7.2.3 Chemical safety glasses or face shield (preferable).

7.3 For known or suspected corrosives, an eyewash station and emergency shower should be located close to the work area, and a two-member crew is required.

7.4 For water matrices (e.g., groundwater, surface water), minimum PPE is safety glasses.

7.5 Consult the MSDS before performing work.

8.0 Preparation of Equipment

8.1 Verify each batch of pH paper versus certified pH buffers or a pH meter that has been calibrated with certified buffers.

8.2 Document verification in the FLB/FN.

9.0 Procedure

9.1 Drum inspection and opening

9.1.1 For drum sampling, particularly drums with unknown contents, examine the drum for its general condition. Note condition in the FLB/FN.

9.1.2 Look for rusting, leaking, bulging, improperly secured cover, and other abnormal conditions.

WARNING: Do not open bulging drums.

9.1.3 Open the drum.

9.1.4 Do not lean over the drum while measuring pH.

9.2 Sampling and Testing

9.2.1 Collect a representative sample.

9.2.2 pH measurements may or may not be taken directly from the source, depending on conditions.

9.2.3 Test the sample with wide range pH paper to determine the approximate pH. Record the results in the FLB/FN.

9.2.4 Select the appropriate narrow range pH paper.

9.2.5 Take two measurements using the narrow range pH paper. Record the results in the FLB/FN.

9.2.6 pH measurements can be used to define a waste as a corrosive or non-corrosive only if the measured values differ

Figure 13-3. Sample SOP for Field pH Measurements Using pH Paper (continued)

from either threshold limit (pH 2 or 12.5) by a full pH unit.

9.2.7 If readings in the ranges of either pH 1–3 or pH 11.5–13.5 are obtained, then the pH should be determined using a meter.

10.0 Quality Control

10.1 All pH determinations must be performed in duplicate.

10.2 Each batch of pH paper must be verified versus certified pH buffers or a pH meter that has been calibrated with certified pH buffers.

11.0 Reference

11.1 SW-846 Test Method for Evaluating Solid Wastes, Method 9041A.

Figure 13-4. Sample SOP for Field pH of Aqueous Samples by Electrometric Measurement

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Field pH of Aqueous Samples by Electrometric Measurement

(Orion Model 230A)

Prep By:
Approved By:
Effective Date:

CAUTION

This Standard Operating Procedure has been prepared as an example for this handbook and may not be specifically applicable to all activities or organizations. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Figure 13-4. Sample SOP for Field pH of Aqueous Samples by Electrometric Measurement (continued)

1.0 Title: Standard Operating Procedure for Field pH of Aqueous Samples by Electronic Measurement.

2.0 Scope and Application

2.1 This method provides an SOP for field pH measurements and is applicable to aqueous samples and multiphasic waste where the aqueous phase constitutes at least 20% of the total volume of the waste. The pH reading of a sample is the logarithm of the reciprocal of the hydrogen ion concentration (hydrogen ion activity) in moles per liter.

3.0 Summary of Method

3.1 The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode. The measuring device is calibrated using a series of standard solutions of known pH.

4.0 Interference

4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or moderate salinity.

4.2 Coatings of oily material or particulate matter can impair electrode response. Remove oily or particulate film before measuring pH.

4.3 Temperature affects both the electrode output and the sample itself. Therefore, instrument calibration and sample analysis should be performed as near to 25°C as possible.

5.0 Equipment

5.1 Orion Research pH Meter Model 230A (or equivalent) or an appropriate portable pH meter with automatic temperature compensation.

5.2 Thermometer, centigrade — calibrated and able to read ± 1 degree.

5.3 ASTM Type I or II water wash bottle.

5.4 Clean wipes.

5.5 Sample collection containers.

5.6 FLB/FN.

6.0 Reagents and Materials

6.1 pH 4.0, 7.0, 10.0 (at 25°C) buffer solutions (buffer solutions to bracket expected sample values).

6.2 pH electrode filling solution (follow manufacturer’s instructions).

6.3 pH electrode storage solution (follow manufacturer’s instructions).

6.4 ASTM Type I or II water.

7.0 Health and Safety Precautions

7.1 Wear appropriate PPE (minimum of chemical safety glasses and appropriately selected gloves).

7.2 Respiratory protection is generally not required for routine samples.

7.3 An eyewash/safety shower should be present, when practical.

8.0 Calibration and Standardization

8.1 Fill the pH electrode with fill solution, and precondition electrodes per the manufacturer's specifications of reference

Figure 13-4. Sample SOP for Field pH of Aqueous Samples by Electrometric Measurement (continued)

11.2.

8.2 Remove the pH electrode from the storage solution, and thoroughly rinse with ASTM Type I or II water. Blot dry with a clean wipe.

8.3 Position the pH probe in a sample of ASTM Type I or II water, and ensure the porous junction of the probe is completely immersed.

8.4 Press the power key until the pH mode indicator is displayed. When the electrode is stable, the READY prompt will be displayed. The pH of the ASTM Type I or II water should be approximately six.

8.5 Remove electrode from ASTM Type I or II water sample, rinse electrode with ASTM Type I or II water, and blot dry.

8.6 Place the electrode in pH 7 buffer, and press the “cal” key. CALIBRATION will be displayed above the main readout, and P1 is displayed in the lower field.

8.7 Wait for a stable pH display, then press the “up arrow” or “down arrow” key and the first digit will start flashing. Scroll until the correct value appears in the first digit. Press “yes”. The second digit will start flashing. Scroll until the correct value appears, then press “yes.” Continue in this manner until all digits have been correctly entered.

8.8 The display will remain frozen for 2 seconds; then, P2 will be displayed in the lower field indicating the meter is ready for the next buffer (4 or 10 buffer).

8.9 Rinse the electrode with ASTM Type I or II water, and place the electrode into the second buffer. Wait for a stable pH display, then enter the correct value.

8.10 The electrode slope (in percent) is then displayed in the main field with SLP in the lower field. The meter automatically advances to MEASURE mode.

8.11 Record the slope in the FLB/FN. The slope shall be from 95% – 105%. If the slope is less than 95% or more than 105%, then recalibrate the pH meter.

8.12 Rinse the probe with ASTM Type I or II water, and place the electrode into the low range pH buffer solution. Record the pH buffer and temperature reading. If the pH is not within ± 0.05 units of the solution value, then recalibrate the pH meter.

8.13 Rinse the electrode with ASTM Type I or II water, and place the electrode into the high range pH buffer solution. Record the pH buffer and temperature reading. If the pH is not within ± 0.05 units of the solution, then recalibrate the pH meter.

8.14 Record calibration date and pH meter identification number in the appropriate FLB/FN.

Note: Press the power key to preserve power.

9.0 Procedure

9.1 Only calibrated equipment shall be used for sample analysis.

9.2 Agitate (without aeration) the sample, and decant an aliquot sufficient for analysis into a clean sample bottle.

9.3 Remove the pH electrode from the storage solution, thoroughly rinse the electrode with ASTM Type I or II water, and blot it dry with a soft tissue.

9.4 Position the electrode in the well-mixed sample solution.

9.5 Press the power key. When the electrode is stable, the READY prompt will be displayed and the temperature-corrected value for the sample will be displayed. Record the pH, sample identification, and sample time in the FLB/FN.

9.6 If elemental analytes are to be determined, then preserve the remainder of the sample by acidifying with nitric acid

Figure 13-4. Sample SOP for Field pH of Aqueous Samples by Electrometric Measurement (continued)

to a pH of ≤ 2.

10.0 Quality Control

10.1 The electrode shall be rinsed between samples.

10.2 Buffers shall be NIST traceable and shall have a current shelf life.

10.3 pH meters shall have a current METCAL (Navy Calibration) calibration sticker.

10.4 Calibration of mercury thermometers shall be checked at least annually against a NIST traceable certified thermometer.

10.5 The calibration of dial-type thermometers shall be checked at least quarterly against a NIST traceable thermometer.

11.0 References

11.1 Standard Methods for the Examination of Water and Wastewater, 21st Edition: Method 4500-H, pH Value.

11.2 Orion Research pH Meter Model 230A Instruction Manual.

11.3 SW-846, Method 9040C, pH Electrometric Measurement

Figure 13-4. Sample SOP for Field pH of Aqueous Samples by Electrometric Measurement (continued)

Data Sheet
pH Meter Standardization

Date of Standardization: ___________________
Time of standardization: ___________________
Analyst: ___________________

Instrument Model: ___________________
Instrument S/N: ___________________
Calibration Exp. Date: ___________________
Buffer Exp. Date: ___________________

Buffer Solution	*Temperature*	*Meter Reading*	*pH Corrected (to 25°C)*
7.0	_______________	___________________	____________________
4.0	_______________	___________________	____________________
10.0	_______________	___________________	____________________

Slope _________

Analyst Signature and Date: ______________________________

Figure 13-5. Sample SOP for Oxidizer Field Testing for Cyanide Samples

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Oxidizer Field Testing for Cyanide Samples

Prep By:
Approved By:
Effective Date:

CAUTION

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Figure 13-5. Sample SOP for Oxidizer Field Testing for Cyanide Samples (continued)

1.0 Title: Standard Operating Procedure for Oxidizer Field Testing for Cyanide Samples.

2.0 Scope and Application

2.1 This SOP covers the procedure needed to test for oxidizers in aqueous samples requiring cyanide analysis. Oxidizers are best removed at time of collection as they destroy/decompose most cyanides, causing a false low result.

3.0 Summary of Method

3.1 A few drops of sample are placed on potassium iodide-Starch test paper (KI starch paper). A “blue” color indicates the presence of oxidizing agents, which may destroy or decompose cyanides. The sample must then be treated with ascorbic acid to neutralize any oxidizing agents.

4.0 Interferences

4.1 None.

5.0 Equipment

5.1 Disposable droppers.

5.2 Sample containers.

5.3 FLB/FN.

6.0 Reagents and Materials

6.1 Potassium iodide -starch test paper (KI starch paper).

6.2 Standard oxidize -potassium dichromate 5%.

6.3 Ascorbic acid, solid.

7.0 Health and Safety Precautions

7.1 Minimum PPE shall be chemical safety goggles (cyanide solutions can be corrosive) and protective gloves. Other PPE will be dictated by the specific hazards of the sampling site (e.g., safety shoes, hard hats).

7.2 On returning from sampling, personnel will wash exposed skin with soap and water.

7.3 Emergency eyewash/safety shower must be present, as appropriate

8.0 Calibration and Standardization

8.1 Take a drop from the potassium dichromate solution (paragraph 6.2) and test the KI starch paper. The strip should turn blue.

9.0 Sampling and Testing

9.1 Collect a representative 1-liter sample.

9.2 With a disposable dropper, put a drop of sample on a strip of KI starch paper. A blue color on the strip indicates that the sample contains oxidizing agents (e.g., chlorine).

9.3 If a blue color is observed, add ascorbic acid, a few crystals at a time, until a drop of sample produces no color change to the KI starch paper.

9.4 Add an additional 0.6 grams of ascorbic acid for each liter of sample.

9.5 Preserve the sample according to the sample preservation guidelines.

Figure 13-5. Sample SOP for Oxidizer Field Testing for Cyanide Samples (continued)

9.6 Record in the FLB/FN that the sample was treated for oxidizer or that the oxidizer test was negative. Also, enter information on the appropriate data sheet.

10.0 Quality Control

10.1 KI starch strips are checked for functionality prior to use.

11.0 References

11.1 SW-846 Test Methods for Evaluating Solid Wastes, Method 9010A.

Figure 13-6. Sample SOP for Field Temperature Determination

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Field Temperature Determination

Prep By:
Approved By:
Effective Date:

CAUTION

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Figure 13-6. Sample SOP for Field Temperature Determination (continued)

1.0 Title: Standard Operating Procedure for Field Temperature Determination.

2.0 Scope and Application

2.1 This method is applicable to drinking, surface waters, domestic and industrial waste, and other various aqueous matrices.

3.0 Summary of Method

3.1 Temperature measurements may be made with any good grade of mercury - filled or dial type centigrade thermometer or thermistor.

4.0 Interferences

4.1 Temperature measurements must be made in the field because temperature is subject to change if there is a time delay in taking the reading.

4.2 Time must be allowed for temperature reading to reach equilibrium (no observed change for 30 seconds).

5.0 Equipment

5.1 Mercury, bimetallic, or electronic thermometers (thermometers should be readable in increments of 1 degree).

5.2 Rinse wastewater container.

5.3 ASTM Type I or II water squirt bottle.

5.4 Clean wipes.

5.5 Rubber gloves.

5.6 FLB/FN.

5.7 PPE.

5.8 Sample containers.

6.0 Reagents and Materials

6.1 ASTM Type I or II water.

7.0 Health and Safety Precautions

7.1 Wear appropriate PPE. As a minimum, safety glasses and protective gloves should be worn.

7.2 Respiratory protection is generally not required for routine samples. However, personnel performing this procedure should be aware of any potential volatile hazards that may require a respirator.

8.0 Procedure

8.1 Ensure thermometer has a current METCAL calibration sticker.

8.2 Immerse the sensor of the thermometer in the material to be tested.

8.3 Observe the temperature reading when equilibrium has been achieved (no change for 30 seconds).

8.4 Record the reading in the appropriate FLB/FN or other appropriate log.

8.5 Remove thermometer from sample.

8.6 Rinse the thermometer with water from the rinse water bottle, catching rinse in the waste container.

Figure 13-6. Sample SOP for Field Temperature Determination (continued)

8.7 Repeat procedure for additional temperature determination, or store thermometer.

9.0 Quality Control

9.1 Ensure thermometers have a current METCAL calibration.

9.2 The calibration of mercury thermometers shall be checked at least annually against a NIST-traceable certified thermometer.

9.3 The calibration of dial-type thermometers shall be checked at least quarterly against a NIST-traceable thermometer.

9.4 Do not use thermometers where the indicating fluid has become separated.

10.0 Reference

10.1 Standard Methods for Examination of Water and Wastewater, 21st Edition: Section 2550 B, Temperature.

Figure 13-6. Sample SOP for Field Temperature Determination (continued)

Field Temperature Data Sheet

Thermometer Control Number: ______________________________

Calibration Expiration Date: ______________________________

Analyst: ______________________________

Remarks, Conditions: ____
____
____
____

Temperature Log

Date/Time	Sample Location	Reading °F / °C	Analyst Initials	Remarks

Reviewed By: ____

Date: ____

Figure 13-7. Sample SOP for Calibration & Maintenance of a Photovac PID

DoD Environmental Sampling and Field Testing Procedure

Standard Operating Procedure

Calibration & Maintenance of a Photovac Photo Ionization Detector

Prep By:
Approved By:
Effective Date:

CAUTION

This Standard Operating Procedure has been prepared as an example for this handbook and may not be specifically applicable to all activities or organizations. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Figure 13-7. Sample SOP for Calibration & Maintenance of a Photovac PID (continued)

1.0 Title: Calibration & Maintenance of a Photovac Photo Ionization Detector (PID)

2.0 Scope and Application

2.1 To calibrate and perform periodic maintenance on a PhotoVac PID Model No. XXXX.

2.2 Calibration is required at the start of each day, and after each time maintenance is performed on the instrument during the day (replacement of bulb or filter, etc.).

3.0 Summary of Method

3.1 Calibration — the measuring device is calibrated using a series of standard gases of known concentration.

3.2 Maintenance — requires the disassembly, replacement of minor components, and the reassembly of the instrument.

Note: Recalibration must be performed after each maintenance operation.

4.0 Equipment

4.1 PID with manufacturer’s instructions.

4.2 Spare battery.

4.3 Battery charge.r

4.4 Spare bulb.

4.5 Spare particulate filter.

4.6 Wrenches, screwdrivers, as required, to disassemble the instrument for maintenance.

4.7 Cotton tipped swabs.

4.8 Clean/New sampling bags.

4.9 FLB/FN.

5.0 Reagents and Materials

5.1 Methanol.

5.2 Cylinder of pure air, or activated carbon filter to make pure air and a sampling pump to fill the sampling bag.

5.3 Cylinder of calibration gas.

6.0 Safety and Health Precautions

6.1 Serious eye damage can result from direct view of UV radiation.

6.2 Calibration gases are pressurized. Handle containers carefully.

6.3 Methanol is flammable.

7.0 Preparation of Equipment for Calibration

7.1 Carefully unpack the instrument and examine it for physical damage that could have occurred in transit.

7.2 The unit’s battery should be charged for 16 hours before use, since the battery can lose its charge during storage.

7.3 Completely empty a flexible air sampling bag (Tedlar® or equivalent) using a vacuum (or sampling) pump.

Figure 13-7. Sample SOP for Calibration & Maintenance of a Photovac PID (continued)

8.0 Procedure for Calibration

8.1 Perform any maintenance operations prior to calibration since disassembly/reassembly requires recalibration.

8.2 Any deviations from the following procedures shall be per the manufacturer's instructions and noted in the FLB/FN.

8.3 Turn the power switch on to activate the unit. The pump should activate, making a continuous low humming sound.

8.4 Check the display for a "LOW BAT" indication (which means the battery should be replaced and/or recharged).

8.5 Allow the unit to operate for at least 2 minutes to come to equilibrium before calibrating.

8.6 Fill the bag with clean air (either "Zero Air" from a cylinder or air that has been passed through a tube of fresh activated charcoal).

8.7 Connect the sample bag to the sampling port of the TIP (inlet port of the instrument). Set the "SPAN" control to maximum, and adjust the "ZERO" knob until the display reads "0". Record the "ZERO" setting in the FLB/FN. Remove the sample bag.

8.8 Fill a sample bag with calibration gas. Record the cylinder manufacturer, lot number, and the label information on the chemicals present in the standard and their concentration in the FLB/FN.

8.9 Attach the sample bag to the sampling port of the TIP (inlet port of the instrument) and adjust the "SPAN" control so that the display reads the same as the concentration on the label of the cylinder. Record both the "SPAN" setting and the display in the FLB/FN. Remove the sample bag.

9.0 Procedure for Replacement of Filter

9.1 Any deviations from the following procedures shall be per the manufacturer's instructions and noted in the FLB/FN.

CAUTION: Certain maintenance procedures may be performed in the field, however, these require the instrument to be recalibrated. Maintenance procedures should be performed only by personnel who have been properly trained.

9.2 The TIP is fitted with a 15-µm cartridge-style filter designed to capture particles that might otherwise be drawn into the detector.

9.2.1 Never use instrument without the filter as the detector can be damaged.

9.2.2 Extended periods of operation in a dusty environment require frequent inspection of the filter for clogging and/or absorption of particles carrying volatile material.

9.2.3 When a filter is clogged, the sensitivity of the instrument decreases since the sample flow-through is decreased. On the other hand, when absorption of the volatile material occurs, a high background reading should be observed and consequently the need for an extreme zero resetting would be required.

9.2.4 The TIP response should be tested with and without the filter and the filter should be replaced if the response varies by more than 10%.

9.3 To access the filter, hold the filter housing firmly at the base with a 9/16-inch wrench and unscrew the 1/8-inch Swagelok® connection for the sample inlet at the top of the housing with another 9/16-inch wrench.

9.4 Replace the filter with the new one.

9.5 Screw the sample inlet connection firmly into place.

9.6 Recalibration (per paragraph 8) is required prior to use.

10.0 Procedure for Lamp Maintenance

10.1 Any deviations from the following procedures shall be per the manufacturer's instructions and noted in the FLB/FN.

Figure 13-7. Sample SOP for Calibration & Maintenance of a Photovac PID (continued)

CAUTION: Certain maintenance procedures may be performed in the field, however, these require the instrument to be recalibrated. Maintenance procedures should be performed only by personnel who have been properly trained.

10.2 The UV lamp should be cleaned if there is a significant loss of sensitivity not due to an obstructed filter.

10.3 Make sure that the unit is switched off before disassembling.

10.4 Refer to the manufacturer's instructions for disassembly and detailed cleaning procedures.

CAUTION: Do not touch the bulb with bare skin, as this can leave an oil film on the bulb, which may damage the equipment.

CAUTION: Under certain circumstances, water can be sucked into the detector. Provided that the water is relatively clean, this may not cause irreparable damage. Usually, the only corrective action needed is to have factory maintenance performed by disassembling and cleaning the unit as soon as possible after the occurrence. Delay will likely result in corrosion of internal parts.

10.5 Clean the lamp with a cotton swab dampened with methanol.

10.6 Be careful not to damage the screens.

10.7 When reassembling the instrument, be careful not to cross-thread the connections or damage the O ring seals.

10.8 After reassembly, perform an operational check of the unit.

10.8.1 If sensitivity is not recovered by cleaning or the lamp will not start, it is likely that a new lamp is required.

10.9 Recalibration per paragraph 8 is required prior to use.

11.0 Quality Control

11.1 Quality control is maintained by strict adherence to these procedures performed by personnel who are trained and familiar with the equipment.

11.2 Only certified calibration gases are used.

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Chapter 14. Guidelines for Requesting Laboratory Testing

14.1. Purpose

This chapter provides guidance for government, or contractors performing work for the government, on requesting laboratory testing and selecting laboratories for environmental sampling. Typically, formal quotes should be requested from multiple laboratories. In rough order of importance, laboratory selection should be based on the following:

- Accreditation/certification status
- Successful performance in related proficiency testing (PT) programs, where available
- Ability to meet project-specific QA/QC requirements
- Capacity to handle the specified number of samples within the required turnaround time
- Cost.

Once selected, communication with the laboratory prior to, during, and after sampling is essential to ensure that reported results will meet regulatory requirements and can be used as intended.

14.2. Scope

This chapter addresses the technical aspects of requesting laboratory testing services and selecting laboratories. Specifically, it contains guidance for preparing requests for quote (RFQ) or scopes of work (SOW). The RFQ or SOW must contain sufficient information to permit the laboratory to understand the requirement, provide a complete and responsive quote, and perform the tests and report the results in accordance with relevant requirements. In the case of projects for which a SAP has been prepared, the laboratory should be provided with a copy of the plan at the time the proposal or quote is solicited. The SOW and project planning documents should be included or referenced in the laboratory's contract.

14.3. Responsibilities

The regulated party (e.g., permit holder or effluent generator) ultimately is responsible for obtaining valid test data. This includes the development of appropriate sampling designs, the collection of representative samples, the performance of the appropriate methods or tests, and the implementation of required health and safety protective measures. Where sampling and testing are to be conducted by contractors, the regulated party is responsible for developing complete and accurate SOWs. The responsibilities of all parties involved in environmental sampling and testing must be clearly identified and agreed upon during project planning.

Typical Responsibilities for Sampling Personnel

- Selecting correct sample locations
- Collecting and preserving samples according to project-planning documents and the permit or regulation
- Labeling samples for proper identification by others
- Documenting any deviations from specified sampling procedures
- Recording all relevant field observations
- Initiating and maintaining sample COC records
- Notifying the laboratory of expected sample arrival dates and times
- Packing and shipping samples in a manner that preserves samples' integrity, maintains the chain of custody, complies with shipping regulations, and ensures timely delivery of the samples to the laboratory.

Typical Laboratory Responsibilities

- Providing prepared containers for sample collection if requested
- Checking samples immediately upon receipt for sample condition and proper preservation
- Notifying the DoD customer in a timely manner of any conditions that have potentially compromised sample integrity
- Properly segregating and storing samples
- Maintaining laboratory chain-of-custody
- Preparing and analyzing samples according to the SOW and project-planning documents

- Evaluating and reporting the results according to specifications
- Advising the customer in a timely manner of any problems, deviations from protocols, or control criteria performance issues
- Providing technical assistance, if requested, for data interpretation.

14.4. Hazards and Safety Precautions

See Chapter 3 for DOT requirements for shipping samples to the laboratory. Samples must be properly labeled, and the outside containers properly marked, so that all persons handling the samples are able to recognize the contents and observe all pertinent safety precautions during sample handling, transfer, storage, and analysis. The laboratory's hazard communication program must comply with 29 CFR 1910.1200.

14.5. Planning

Ideally, a DoD Chemist should be consulted during planning, to assist in developing the RFQ or SOW. At a minimum, the DoD Chemist should review the RFQ or SOW to ensure it is technically adequate. The following information should be determined during planning and included in the RFQ or SOW.

1. **Regulatory Requirements.** Describe the relevant regulation and purpose for testing (e.g., compliance monitoring under NPDES, drinking water testing, RCRA groundwater monitoring)
2. **Parameter(s) and Analytical Methods.** List all parameters to be analyzed and reported, and specific methods (e.g., sample cleanup, preparation, extraction, and analysis) to be used. For further guidance on this topic, see Section 14.6.
3. **Sample Descriptions and Sample Handling Requirements.** Provide a summary of the number and descriptions of sample matrices to be submitted for analysis (e.g., groundwater, soil, and wastewater). Describe any known or suspected toxic or hazardous samples. If the samples consist of multiple phases, this should be noted as well. Describe the number and type of sample bottles and sample preservation that will be used. Note whether the sampling team or the laboratory is to supply sample containers and preservatives.
4. **Laboratory Qualifications.** Describe all project-specific requirements for laboratory qualifications, including certification or accreditation requirements and participation in specific PT studies. Section 14.7 describes the minimum qualifications laboratories must meet in order to be eligible to perform testing for DoD.
5. **QA/QC.** Describe any project-specific quality assurance and quality control requirements and measurement performance criteria that differ from method QA/QC requirements. If applicable, include copies of the SAP or QAPP.
6. **Data Reporting Requirements.** Describe the content and format requirements for laboratory reports, including both hard-copy reports and electronic data deliverables (EDDs). For each parameter to be reported, list the required reporting units (e.g., percent, mg/L, ppm, ppb, dry weight/wet weight basis) and quantitation limits. Quantitation limits must be at or below compliance limits specified in the permit or regulation.
7. **Project Schedule.** Provide a schedule or timeline showing expected sampling dates, expected sample delivery dates, required sampling and analysis hold times, and required analytical turnaround time. For more guidance on this topic, see Section 14.10.

Roles for the DoD Chemist

A DoD Chemist should be consulted during project planning to assist in developing the RFQ or SOW. At a minimum, the chemist should review the RFQ or SOW to ensure it is technically adequate. A DoD Chemist also can:

- Develop or review project-specific performance standards and DQOs
- Verify the appropriateness of sampling and analytical methods

- Provide oversight for development and correct implementation of the SAP
- Notify the Contracting Officer of potential laboratory non-conformance issues, including prohibited practices
- Monitor corrective action, where necessary
- Perform data review.

14.6. Parameters and Analytical Methods

The permit or regulation typically prescribes the analytical parameters and methods to be used. The parameters tested and reported should be limited to those required in the scope of the regulation or permit. There are three primary criteria for method selection: 1) its use must be allowed under the permit or regulation, 2) it must be appropriate for the type of matrix being sampled, and 3) it must be capable of generating quantitative results with adequate data quality at or below the regulatory limit.

Once a facility has selected and begun using a specific method, it should exercise caution before changing either laboratories or methods, as this can affect the comparability of analytical results from one monitoring event to the next. To help evaluate data comparability between laboratories or methods, the permit holder may employ the analysis of split samples by two or more qualified laboratories.

In general, analytical methods used for regulatory compliance should be performed as written, and the laboratory must adhere to all method-specified quality control criteria. Modifications to methods specified in permits are allowed only if: 1) they are necessary to the successful analysis of regulated parameter in a particular sample matrix, and 2) the permit writer agrees to the change(s) prior to implementation. Care must be taken to ensure method modification does not alter the sample chemistry.

Most published methods accommodate some degree of flexibility, and in those cases, the laboratory may use discretion for specific steps in the method where allowed. In any case, the exact procedure used must be documented in the laboratory's SOP for that method.

The following paragraphs discuss the selection of parameters and methods for some common types of compliance monitoring.

Wastewater. Parameters controlled under the NPDES provisions of the CWA are listed in the NPDES permit application, Form 2C. Testing requirements including frequency, sample location, sampling method, and QC requirements, also are described in the permit.

The approved test methods for wastewater, along with sample holding times and preservation, are listed in 40 CFR Part 136. In March 2007, EPA proposed a number of new and revised analytical methods used under both the CWA and Drinking Water regulations. The proposed changes, found in the Federal Register, March 12, 2007 (Volume 72, Number 47), include the following:

- Additional new methods and updated versions of currently approved methods
- Revised method modification and analytical requirements
- Withdrawal of certain outdated methods
- Changes to sample collection, preservation, and holding time requirements.

Drinking Water. The water supplier's operating permit describes the test parameters, analytical methods, sampling methods, sampling frequency and compliance limits. The primary reference for drinking water methods is 40 CFR Part 141.21-142.30 and *Manual for the Certification of Laboratories Analyzing Drinking Water* (Fifth Edition, January 2005). As discussed above under Wastewater, the EPA Office of Water proposed a number of new and revised analytical methods (Federal Register, March 12, 2007 (Volume 72, Number 47)). Other standard methods may be acceptable, including Standard Methods for the Examination of Water and Wastewater and ASTM Water and Environmental Technology Methods.

Solid and Hazardous Waste. Under the RCRA regulations, the waste generator is responsible for correctly characterizing its waste and may use knowledge of the waste in the process for selecting the test parameters. Solid wastes are classified as hazardous if they 1) are specifically listed in 40 CFR Part 261, 2) are deemed hazardous by the generator based on process knowledge, or 3) meet the characteristics of a hazardous waste specified in 40 CFR Part 261. Waste sampling is described in Chapter 10 of this handbook. The generator must understand and comply with all regulations found in 40 CFR Parts 260 to 261 before selecting methods for waste characterization. RCRA waste characterization methods are published in EPA SW-846 Test Methods for Evaluating Solid Waste, as updated.

Groundwater. The parameters and analytical methods for potable water testing are based on the drinking water regulations found in 40 CFR Part 141. For landfill monitoring, the landfill development permit specifies sampling methods, sampling frequency, whether to use filtered or unfiltered samples, purge methods, compliance limits, and test parameters. 40 CFR Part 265.90, Subpart F – Groundwater Monitoring contains regulations for groundwater monitoring at landfills. The regulations for landfill leachate monitoring are found in the solid waste landfill regulation in 40 CFR Part 258.

Underground Storage Tanks. The primary reference for selecting methods for analyzing the contents of USTs and leaking underground storage tanks (LUST) is 40 CFR Part 280. The methods for removal and additional monitoring are specified by state agencies for soil and groundwater monitoring. Additional information on sampling the contents of USTs is provided in Chapter 10 of this handbook.

Dredging Operations. Sampling and testing requirements for the purpose of obtaining dredging permits will vary based on past and future uses of the water body, regulatory requirements, and known contaminants. For the purpose of determining the proper handling of dredging spoils, sediment monitoring parameters can include RCRA hazardous waste characteristics, TSCA parameters (e.g., PCBs, dioxins and furans), and NPDES parameters. For the purpose of monitoring the impacts of dredging activities on water quality, dredging permits may require monitoring of surface water as well as sediments.

Note: When selecting or modifying methods, more extensive discussion is available in the EPA guidance document, Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring, EPA 821-B-93-001, June 1993.

14.7. Laboratory Selection

Laboratories performing testing in support of the Defense Environmental Restoration Program must be accredited in accordance with the DoD ELAP.

Laboratories must possess any required state or host nation certification and/or be accredited for each applicable test method, by a nationally recognized laboratory accreditation body (e.g. the National Environmental Laboratory Accreditation Program or NELAP), compliant with International Organization for Standardization (ISO/IEC) 17011:2004. All laboratories must demonstrate the ability to generate acceptable results from the analysis of PT sample(s), subject to availability, using each applicable method in the specified matrix. Upon request, laboratories must make available to DoD the results of all PT samples analyzed by the laboratory during the period of performance. The laboratory shall make appropriate documentation available to the DoD Chemist/Quality Assurance Manager (QAM). All laboratories are subject to on-site assessments by authorized DoD representatives. The laboratory shall notify DoD in writing of any change in laboratory certification or accreditation status within 30 calendar days of the change. This written notification requirement applies, but is not limited to, suspension or revocation of DoD ELAP accreditation.

Laboratories shall have a documented program to prevent improper, unethical, or illegal actions and must not engage in any prohibited practices. The laboratory must have the appropriate accreditation for the testing to be performed. The laboratory must not commence testing until DoD has accepted the laboratory's quality systems documentation.

14.8. QA/QC

Detailed information on QA/QC, including the systematic planning process, development of DQOs, and preparation of SAPs is provided in Chapter 2 of this handbook. Laboratories must follow all method-specified quality assurance and quality control procedures and observe method- or project-specified performance criteria.

14.9. Reporting

Laboratory reports must include a narrative describing the samples received, analyses, quality control exceedances, and any unusual observations made during analysis. The use of subcontractors must be identified in the primary laboratory's final report. The minimum recommended contents of laboratory reports for compliance sampling include the following:

In the absence of client specified reporting criteria, the reporting requirements outlined below shall be used for hard-copy data reports from the laboratory. They are divided into mandatory requirements for all printed data reports, and optional requirements. Optional reporting requirements are those that may be required by a specific project, depending upon the needs of the project. The following elements are required: cover sheet, table of contents, case narrative, analytical results, sample management records, and QA/QC information. Information for third-party review may be required depending on project-specific requirements or the method being used. The requirements below do not dictate what records the laboratory should maintain.

1. **Cover Sheet.** The cover sheet shall 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
   - Unique identification of the report (such as serial number)
   - Client name and address
   - Project name and site location
   - Statement of data authenticity and official signature and title of person authorizing report
   - Amendments to previously released reports that clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report
   - Total number of pages.
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 shall be included for this purpose.
3. **Case Narrative.** A case narrative shall be included in each report. The purpose of the case narrative is to describe any abnormalities and deviations that may affect the analytical results. It is also used to summarize any issues in the data package that need to be highlighted for the data user to help them assess the usability of the data. The case narrative shall provide:
   - A table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical methods were performed. If multiple laboratories performed analyses, the name and location of each laboratory should be associated with each sample.
   - A list of samples that were received but not analyzed
   - A description of extractions or analyses that are performed out of holding times
   - A definition of all data qualifiers or flags used

- Identification of deviations of any calibration standards or QC sample results from appropriate acceptance limits and a discussion of the associated corrective actions taken by the laboratory
- Identification of samples and analytes for which manual integration was necessary
- Appropriate notation of 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.)
- Identification of numerical results outside of limits of quantitation.

4. **Analytical Results.** The results for each sample shall contain the following information at a minimum (information need not be repeated if noted elsewhere in the data package):

- Project name and site location
- Field sample ID number as written on custody form
- Laboratory sample ID number
- Matrix (soil, water, oil, etc.)
- Date and time sample extracted or prepared
- Date and time sample analyzed
- Method numbers for all preparation, cleanup, and analysis procedures employed
- Analyte or parameter
- Method reporting limits and method limits of quantitation (at or above the low-level standard concentration) adjusted for sample-specific factors (e.g., aliquot size, dilution/concentration factors, moisture content)
- All samples and analytes for which manual integration occurred, including the cause and justification
- Limits of detection and/or method detection limits
- Analytical results with correct number of significant figures
- Any data qualifiers assigned
- Concentration units
- Dilution factors
- Any dilutions or concentrations for all reported data, and if neat or less diluted results are available, recorded and reported data from both runs
- Percent moisture or percent solids (all soils are to be reported on a dry weight basis).

The following information is optional but may be required site-specifically:

- Laboratory name and location (city and state)
- Sample description
- Sample preservation or condition at receipt
- Date and time sample collected
- Date sample received
- Sample aliquot analyzed
- Final extract volume
- Chemical Abstract Service (CAS) numbers
- Statements of the estimated uncertainty of test results.

5. **Sample Management Records.** These types of records include the documentation accompanying the samples, such as:

- COC records
- Shipping documents
- Records generated by the laboratory which detail the condition of the samples upon receipt at the laboratory (e.g., sample cooler receipt forms)
- Telephone conversation records associated with actions taken or quality issues
- Description of the sampling procedures if the laboratory collected the sample.

6. **QA/QC Information.** The minimum internal QC data package must include the following:

- MS recovery

- Relative percent difference (RPD) of required duplicates
- Laboratory Control Sample (LCS) recoveries
- In-house LCS control limits, if they exceed DoD limits
- Surrogate recoveries (organics)
- Tracer recoveries (radiochemical)
- Method blank results
- Preparation, analysis, and other batch numbers
- QC acceptance criteria for MS, LCS, surrogates, etc.
- Spike concentrations for MS, LCS, surrogates, etc.

7. **Information for Third-Party Review.** The information listed below is required if third-party (from outside the laboratory) data validation or verification is to be performed. This information is therefore optional and is provided only when the project-specific or regulatory requirements specify that a third-party review will occur.
    - Calibration data from the initial calibration curve
    - Initial calibration verification (ICV)
    - Continuing calibration verification(s) (CCV)
    - Performance standards analyzed in conjunction with the test method (e.g., tuning standards, degradation check standards, etc.)
    - Preparation, analysis, and other batch numbers
    - Raw data (e.g., chromatograms, mass spectrum results)
    - MS, if applicable (includes spike target concentration levels, measured spike concentration, and calculated recoveries)
    - RPD of required duplicates (e.g., MSD, LCS duplicate, matrix duplicates (MD), and field duplicates)
    - Method blank results
    - LCS recoveries[1]
    - Surrogate recoveries (organics)
    - Serial dilutions (SD) percent difference (metals)
    - Post-digestion spikes (PDS) recovery (metals)
    - Project action levels, DQOs, Measurement Quality Objectives (MQOs), and associated acceptance criteria
    - Supporting documentation (e.g., run logs, sample preparation logs, standard preparation logs).

In addition, the data package for third-party review may include summary forms from detection limit studies. The data validation guidelines for performance-based methods established in other DoD guidance on data review and data validation, EPA national functional guidelines, EPA 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.

If compliance limits are exceeded or unusual data results are reported by the laboratory, further data review may be initiated.

It is recommended that at a minimum, the laboratory report be reviewed to make sure all samples were analyzed for the correct parameters, using the correct methods, within the specified holding times, and that the report conforms to specifications for content and format.

14.10. Project Schedule Considerations

The time from sample collection until the start of sample preparation is the holding time referred to in the regulations. In general, the time of sample collection time refers to the time when the sample first leaves its natural environment. EPA has established holding times to maintain sample integrity and stability and ensure representative analytical results. Samples must be preserved and packaged according to method specifications and received by the laboratory in time to permit the preparation and analysis within the required holding times.

Analytical turnaround time is the time between sample receipt and delivery of the final report to the customer. Table 14-1 lists the approximate minimum time needed to prepare and analyze certain types of samples. When estimating the minimum turnaround times possible for project planning purposes, add 1 day for sample receipt, preservation check, and sample log-in at the start of processing and 1 day at the end for data and QC review.

Table 14-1. Minimum Analytical Processing Time

Parameter	Preparation Time	Analysis Time
BOD	0.5 day	5 days
Solids	0.5 day	1 day
Wet chemistry	1 day	0.5 day
Metals, dissolved	0.5 day	0.5 day per method
Metals, total acid digestion	0.5 days water 1 day solids	0.5 day per method
Volatiles	0.5 day	1 day
Semi-volatile organics	2 days	1 day
Pesticides	1 day	1 day
TCLP-metals	2 days	0.5 day per method
TCLP-ZHE-volatiles	1 day	1 day
TCLP-semivolatiles	2 day	1 day
Radionuclides	2 days	1 to 15 days

Appendix A. Sampler/Sampling Recommendations and Strategies

Table A-1. Recommended Samplers for Various Types of Waste

(See Note 1 below)

Waste Type	Recommended Sampler	Limitations
Liquids, sludges, and slurries in drums, vacuum trucks, barrels and similar containers	COLIWASA, Open Tube (Thief), Stratified sample (Thief)	Not for containers greater than 1.5 m (5 ft) deep
	a) Plastic	Not for wastes containing ketones, nitrobenzene, dimethylformamide, mesityl oxide, or tetrahydrofuran
	b) Glass	Not for wastes containing hydrofluoric acid and concentrated alkali solutions
	c) Teflon®	None
Liquids, sludges, and slurries in drums, vacuum trucks, barrels, and similar containers	Peristaltic Pump	For containers more than 1.5 m (5 ft) deep
Liquids and sludges in ponds, pits, or lagoons	Pond sampler	Cannot be used to collect samples beyond 3.5 m (11.5 ft) Dip and retrieve sampler slowly to avoid bending the tubular aluminum handle
Powdered or granular in bags, drums, barrels, and similar containers	a) Grain sampler	Limited application for solids sampling moist and sticky solids with a diameter 0.6 cm ($^1/_4$ in)
	b) Sampling trier	May incur difficulty in retaining core sample of very dry granular materials during sampling
Dry wastes in shallow containers and surface soil	Trowel or scoop	Not applicable to sampling deeper than 8 cm (3 in) Difficult to obtain reproducible mass of samples
Waste piles	Waste pile sampler	Not applicable to sampling solid wastes with particle dimensions greater than half the diameter of the sampling tube
Solid deeper than 8 cm (3 in)	a) Soil auger	Does not collect undisturbed core samples
	b) Veihmeyer sampler	Difficult to use on stony, rocky, or very wet soil
Waste in storage tanks	a) Weighted bottle sampler	May be difficult to use on very viscous liquids
	b) Bacon Bomb sampler	Volume restriction 1 L maximum
	c) Kemmerer sampler	May need extra weight

Note 1: These are only recommendations. Consult the applicable regulations to confirm sampler selection recommendations.

Table A-2. General Sampling Strategies

(See Note 1 below)

Sample Source	Matrix	Sampling Strategy
Drum	Liquid or solid	Collect at least one sample from each drum through the bung on top. If contents are stratified, collect at least one sample from each stratum.
Vacuum truck, storage tank, process vessel	Liquid or solid	Collect at least one sample from each compartment in tank, through open hatch. If contents are stratified collect a sample from each stratum.
Barrel, fiberdrum, bucket, sack, bag	Solid (powder or granular)	Collect at least one sample from each container, through the top or fill opening. Withdraw samples through the center of each container to a point diagonally opposite the point of entry.
Soil	Soil	Random: Divide area into a grid. Grid size is determined based on statistical considerations. Collect at least one sample at each grid node or choose locations using a random numbers table. Depth of collection may vary depending on soil type, parameters and site history. Biased: collect stained soils or soils suspected to be contaminated. Obtain as many samples as necessary to characterize different areas of site. (Note 2)
Waste piles	Solid	Divide pile into a grid and collect sample at the nodes, obtaining a core of 1–2 feet in length at each node. The size of the piles will depend on the project's data quality objectives. (Note 2)
Pond, lagoon	Liquid or sludge	Divide surface into grid. Grid size is determined based on statistical considerations. Collect at least one sample at each node. If material is stratified, collect a sample from each stratum at each node.
Surface water [**Note:** Related to identifying the extent of spills or risk assessments. Not applicable to SDWA or CWA monitoring.]	Liquid	Pond or lake: Establish grid based on statistical considerations, collect samples at nodes. Depending upon objectives, samples may be obtained at the surface, mid-depth or bottom or any combination thereof. Sample should be collected at mid-depth in a channel. Additional samples may be required if multiple discharges and additional streams are present, or for delineation of contamination. (Note 3)
Sediment [**Note:** Related to identifying the extent of spills or risk assessments. Not applicable to SDWA, CWA or ecological monitoring.]	Solid	Pond or lake: Establish grid based on statistical considerations, collect samples at nodes. Stream or river: Collect at least three samples, one downstream, one upstream, and one adjacent to site or at a point of discharge. Additional samples may be required if multiple discharges and additional streams are present. For delineation of contamination, additional discrete downstream samples, transecting the stream and/or at various depths may be employed. (Note 4)

Note 1: These are only recommendations. Consult the applicable regulations to confirm strategy recommendations.
Note 2: Refer to *EPA QA/G5S* for guidance on designing statistically relevant sampling plans.

Note 3: Refer to the USGS's *National Field Manual for the Collection of Water-Quality Data* for additional guidance on collecting surface water samples.

Note 4: Refer to the USGS's *Guidelines for Collecting and Processing Samples of StreamBed Sediment for Analysis of Trace Elements and Organic Contaminants for the National Water Quality Assessment Program* for additional guidance on collecting surface water samples.

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Appendix B. Requirements for Sample Containers, Preservation, and Holding Times

Table B-1 below specifies the required sample containers, preservation, and holding times for test samples. Each column is organized by the terms defined below.

Note: The information provided in Table B-1 represents the sample handling requirements as they exist in the stated method. For the most up-to-date and accurate information, refer to the applicable reference document.

Note: A sampling plan is to be prepared and reviewed with the lab and, in some cases, the regulator prior to starting any sampling/testing operation. Local or state regulations may supersede these requirements.

Parameter. The testing parameters must be specified in the SAP.

Sample Containers. Collection of the size and number of sample containers specified in the following table will assure that the laboratory receives enough sample material to perform the required analyses. Additional sample containers may be required for laboratory QA tests; see Appendix C for details on QA/QC sample requirements.

Container cleaning procedures are found in the test method, laboratory quality program, or regulatory program guidance documents. References for container cleaning procedures and additional container information for RCRA sampling can be found in *U.S. EPA OSWER directive 9240.0-05, Specifications and Guidance for Contaminant-Free Containers, December 1992(http://www.epa.gov/region1/oeme/ATTACHD.pdf)* SDWA sampling containers and cleaning procedures can be found in the *Manual for the Certification of Drinking Water Laboratories, Fifth Edition January 2005*. The laboratory can furnish containers of the required size and cleanliness.

Note: VOA vials are to have Teflon®-faced silicone septum caps. All other jars and bottles are to have Teflon®-lined caps.

Preservation. Some samples must be preserved before shipment to the laboratory.

- Preserve samples immediately upon sample collection
- Cooling to ≤ 6°C can be accomplished by placing sample containers in an insulated plastic shipping cooler (Coleman picnic cooler or equal) along with plastic bags of ice
- Other preservation procedures must be specified completely in the SAP.

Filling Instructions. VOA vials with septum caps are to be filled completely with no headspace or air pockets. For all other containers, leave adequate headspace in containers to allow for thermal expansion of the sample material and mixing of sample.

Holding Time. Samples should be analyzed as soon as possible after collection. Many samples are not stable for lengthy periods following collection, so daily shipment to laboratories is very important. The holding times listed in the following table are the maximum amount of time that the samples may be held before analysis from time of collection and still be considered valid. Samples exceeding these holding times are not valid for compliance and must be retaken.

Table B-1. Requirements for Sample Containers, Preservation, and Holding Times

(See Note 1 below)

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
Alkalinity	Drinking Water	200-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	14 days	SM2320
Alkalinity	Wastewater	200-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	14 days	EPA 310; SM2320
Ammonia as Nitrogen	Stormwater, Surface water, Wastewater	1-L glass or polyethylene bottles	H_2SO_4 to pH < 2; cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA 350; SM4500-NH_3
Asbestos	Drinking Water	2x 1-L glass or polyethylene bottles	Cool to ≤ 6°C.	Leave ample air space in the bottle (at least 2.5 cm).	48 hours	EPA 100
Biochemical Oxygen Demand (BOD_5)	Stormwater, Surface water, Wastewater	1-L glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	SM5210
Bromide	Solid and Liquid Waste	100-mL glass or polyethylene bottles	None required.	Fill to neck of bottle.	As soon as possible	SW-846 Methods 6500, 9056, 9211
Bromide	Wastewater	100-mL glass or polyethylene bottles	None required.	Fill to neck of bottle.	28 days	EPA 300; SM4110
BTEX	Soils and Sludges	2x 40-mL vials *(Note 4)*	Per preparative method. See SW-846 Methods 5035, 5021, 5031, 5032.	Fill completely, no headspace.	14 days	Speciated results from VOC analysis: SW-846 Method 8260, 8015, 8021
BTEX	Wastewater	2x 40-mL vials *(Note 4)*	Cool to ≤ 6°C. If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Adjust pH to 2 with HCl.	Fill completely, no headspace.	14 days	Speciated results from VOC analysis *(Note 6)*: EPA 602, 624, 1624
Carbonaceous Biochemical Oxygen Demand ($CBOD_5$)	Surface water, Wastewater	1-L glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	SM5210
Chemical Oxygen Demand (COD)	Stormwater, Surface water,	100-mL glass bottles	H_2SO_4 to pH < 2;	Fill to neck of bottle.	28 days	EPA 410; SM5220

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
	Wastewater		Cool to ≤ 6°C.			
Chloride	Drinking Water	100-mL glass or polyethylene bottles	None required.	Fill to neck of bottle.	28 days	EPA 300; SM4110, SM4500-Cl^-
Chloride	Wastewater	100-mL glass or polyethylene bottles	None required.	Fill to neck of bottle.	28 days	EPA 300; SM4110; SM4500-Cl^-
Chloride	Groundwater, Other aqueous waste	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SW-846 Methods 6500, 9056, 9212, 9250, 9251, 9253
Chlorinated Acids	Drinking Water	2x 1-L amber glass bottles fitted with screw caps lined with Teflon®	If chlorine is present, add 0.008% $Na_2S_2O_3$. Seal bottle and shake vigorously for 1 minute. Cool to ≤ 6°C, away from light.	Fill to neck of bottle.	Extract within 14 days; analyze within 28 days of extraction	EPA 515
Chlorinated Acids	Wastewater	2x 1-L amber glass bottles fitted with screw caps lined with TFE-fluorocarbon	If chlorine is present, add 0.008% $Na_2S_2O_3$. Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	EPA 625, 604
Chlorine, Residual, Free	Drinking Water	500-mL glass or polyethylene bottles	NA; analyze immediately.	None.	15 minutes	SM4500-Cl^-
Chlorine, Residual, Total	Drinking Water	500-mL glass or polyethylene bottles	NA; analyze immediately.	None.	15 minutes	EPA 330; SM4500-Cl^-
Chlorine, Residual, Total	Wastewater	500-mL glass or polyethylene bottles	NA; analyze immediately.	None.	15 minutes	SM4500-Cl^-
Coliform, Fecal	Drinking Water	120-mL borosilicate glass or plastic bottles, pre-sterilized plastic bags	If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Cool to < 10°C	Leave ample air space in the bottle (at least 2.5 cm).	6 hours	SM9221, 9222

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Coliform, Fecal	Surface water and Wastewater	120-mL glass or plastic bottles, pre-sterilized plastic bags	If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Cool to < 10°C.	Leave ample air space in the bottle (at least 2.5 cm).	6 hours	SM9221, 9222
Coliform, Total	Drinking Water	120-mL glass or plastic bottles or pre-sterilized plastic bags	If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Cool to ≤ 10°C.	Leave ample air space in the bottle (at least 2.5 cm).	6 hours	SM9221, 9222, 9223
Coliform, Total	Surface water, Storm water	120-mL glass or plastic bottles or pre-sterilized plastic bags	If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Cool to < 10°C.	Leave ample air space in the bottle (at least 2.5 cm).	6 hours	SM9221, 9222
Conductivity (Specific Conductance)	Drinking Water	500-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SM2510
Conductivity (Specific Conductance)	Groundwater	100-mL plastic and glass containers	None required.	None.	Analyze immediately	SW-846 Method 9050
Corrosivity	Solid and Liquid Waste	1-L polyethylene jar	None required.	Fill per sampling plan.	None listed	SW-846 Method 1110
Cyanide	Drinking Water	1-L glass or polyethylene bottles	NaOH to pH > 12. Cool to ≤ 6°C. If residual chlorine is present, add small amounts of $Na_2S_2O_3$ solution (0.02 g/L) until oxidizers are neutralized. If other oxidizing agents are suspected, refer to Method 9010C for preservation.	Fill to neck of bottle.	14 days	EPA 335; SM4500-CN^-

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
Cyanide	Groundwater, Other aqueous waste	1-L glass or polyethylene bottles	50%NaOH to pH >12. If oxidizing agent is present, add 0.06 g ascorbic acid per L. Cool to ≤ 6°C.	Fill to neck of bottle.	14 days	SW-846 Method 9010, 9012, 9213
Cyanide	Solid and other non-aqueous	100 g in a glass jar	Cool to ≤ 6°C.	None.	14 days	SW-846 Method 9013
Cyanide	Storm water, Wastewater	1-L glass or polyethylene bottles	50% NaOH to pH >12. If an oxidant is present, add appropriate reducing agent as applicable to the method. Cool to ≤ 6°C. If other oxidizing agents are suspected, refer to Method 9010C for preservation.	Fill to neck of bottle.	14 days (48 hours if not treated for reducing agents)	EPA 335; SM4500-CN$^-$
Dioxins and Furans (PCDDs, PCDFs)	Groundwater	4 x 1-L amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 0.008% $Na_2S_2O_3$. If pH is greater than 9, adjust to pH 7-9 with sulfuric acid.	Fill to neck of bottle.	Extract within 30 days; analyze within 45 days of extraction	SW-846 Methods 8280, 8290
Dioxins and Furans (PCDDs, PCDFs)	Sediment, Soils, and Sludges	250-mL wide mouth glass containers with Teflon® lined lids	Cool to ≤ 6°C and keep in the dark.	Fill to neck of bottle.	Extract within 30 days; analyze within 45 days of extraction	SW-846 Methods 8280, 8290

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Dioxins and Furans (PCDDs, PCDFs)	Wastewater	4 x 1-L amber glass bottles with Teflon® lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 0.008% $Na_2S_2O_3$. If pH is greater than 9, adjust to pH 7-9 with H_2SO_4.	Fill to neck of bottle.	1 year	EPA 1613B
DBPs (Bromate)	Drinking Water	40-mL amber glass vials	EDA to produce 50 mg/L in sample. Store at ≤ 6°C in the dark.	Fill completely, no headspace.	28 days	EPA 300, 317, 326
DBPs (Chlorite)	Drinking Water	40-mL amber glass vials	EDA to produce 50 mg/L in sample. Store at ≤ 6°C in the dark.	Fill completely, no headspace.	14 days (must arrive at the laboratory w/in 48 hours of collection at 6°C or less)	EPA 300, 317, 326
DBPs (HAA5)	Drinking Water	120-mL amber glass vials with TFE-lined caps	Crystalline or aqueous NH_4Cl to produce 100 mg/L in sample. Store at 4°C in the dark.	Fill completely, no headspace.	28 days	EPA 552; SM6251
DBPs(Trihalomethanes)	Drinking Water	2x 60-mL glass vials with Teflon® faced silicone septum	Cool to ≤ 6°C. Samples must contain either 100 mg/L ammonium chloride or $Na_2S_2O_3$ as a dechlorinating agent. Refer to selected method for preservation requirements.	Fill completely, no headspace.	28 days	EPA 551, 502, 524
Dissolved Oxygen, Probe	Groundwater, Surface water and Wastewater	300-mL glass stoppered BOD bottles	None required.	Fill to neck of bottle with minimal aeration.	15 minutes	SM4500-O

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Fluoride	Drinking Water	100-mL polyethylene	None required.	Fill to neck of bottle.	28 days	EPA 300, SM4110, SM4500-F^-
Fluoride	Wastewater	100-mL polyethylene	None required.	Fill to neck of bottle.	28 days	EPA 300, SM4110, SM4500-F^-
Fluoride	Groundwater, Other aqueous waste	300-mL polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SW-846 Method 6500, 9056, 9214
Hardness	Drinking Water	100-mL glass or polyethylene bottles	HNO_3 or H_2SO_4 to pH < 2; Cool to ≤ 6°C.	Fill to neck of bottle.	6 months	EPA 130
Hardness	Wastewater, Surface water	100-mL glass or polyethylene bottles	HNO_3 or H_2SO_4 to pH < 2; Cool to ≤ 6°C.	Fill to neck of bottle.	6 months	EPA 130; SM2340
Herbicides	Sediment, Soils, and sludges, Other non-Aqueous, Solid Waste (concentrated)	250-mL wide mouth glass container with Teflon®-lined lid	Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 14 days; analyze within 40 days of extraction	SW-846 Methods 8151, 8321
Herbicides	Groundwater, Other aqueous waste	4 x 1-L amber glass bottles with Teflon®- lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SW-846 Methods 8151, 8321
Herbicides, TCLP extract *(Note 7)*	Solid Waste	Three 1-L wide mouth amber glass bottles with Teflon® - lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	14 days from field collection to TCLP extraction; 7 days from TCLP to preparative extraction; analyze within 40 days of preparative extraction	SW-846 Methods 1311, 8151, 8321

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
Ignitability	Solid and Liquid Waste	250-mL amber wide mouth glass jar or wide mouth polyethylene bottle	None listed. Review sampling plan for non-aqueous wastes.	Fill with no headspace when volatiles are suspected. Check sampling plan.	Not established	SW-846 Method 1010, 1020, 1030; ASTM D-93-79, D-93-80, D3278-78
Metals (Lead and Copper)	Drinking Water	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	EPA 200, SM3113
Metals (Lead and Copper)	Groundwater, Other aqueous waste	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SW-846 Methods 6010, 6020, 6200, 6800, 7000, 7010
Metals (Lead and Copper)	Solid waste	250-mL glass jar	None required.	Fill to neck of bottle.	6 months	SW-846 Methods 6010, 6020, 6200, 6800, 7000, 7010
Metals (Lead and Copper)	Wastewater	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SM3111; EPA 200; SM3113, SM3120
Metals, Calcium	Drinking Water	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	EPA 200, SM3500-Ca, SM3111; SM3120
Metals, Calcium	Wastewater	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SM3111, SM3120, SM3500-Ca
Metals, Calcium	Groundwater, Other aqueous waste	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SW-846 Method 6010, 6020, 6200, 6800, 7000

Parameter	Matrix	Sample Containers *(Note 2)*	Preservation	Filling Instructions	Holding Time	Method *(Note 3)*
Metals, Calcium	Solid waste	250-mL certified metal-free glass or polyethylene bottles	None required.	Fill to neck of bottle.	6 months	SW-846 Method 6010, 6020, 6200, 6800, 7000
Metals, Dissolved	Groundwater	1-L certified metal-free glass or polyethylene bottles	Filter through 0.45-µm filter. HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SW-846 Method 6010, 6020, 7000, 7010
Metals, Dissolved	Stormwater and Wastewater	1-L certified metal-free glass or polyethylene bottles	Filter through 0.45-µm filter. HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	EPA 200; SM3113, SM3120
Metals, Hexavalent Chromium (Cr^{+6})	Groundwater, Other aqueous waste	500-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	24 hours	SW-846 Methods 6800, 7195, 7196, 7197, 7198, 7199
Metals, Hexavalent Chromium (Cr^{+6})	Soils and Sludges	250-mL glass jar	Cool to ≤ 6°C.	Fill to neck of bottle.	30 days	SW-846 Methods 6800, 7195, 7196, 7197, 7198, 7199
Metals, Hexavalent Chromium (Cr^{+6})	Wastewater	1-L certified metal-free glass or polyethylene bottles	NaOH to pH of 9.3-9.7; Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA 218; SM3111C, SM3500-Cr
Metals, Mercury	Drinking Water	250-mL certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2. Unpreserved samples may be sent to the laboratory within 2 weeks, then acid preserved and held 16 hours before analysis.	Fill to neck of bottle.	28 days	EPA 245; SM3112
Metals, Mercury	Groundwater, Other aqueous waste	500-mL glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	28 days	SW-846 Method 7470

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
Metals, Mercury	Sediment, Solid, Semi-solid waste	250-mL glass jar	Cool to ≤ 6°C	Fill to neck of bottle.	28 days	SW-846 Methods 7470, 7471, 7474
Metals, Mercury	Wastewater and Storm water	500-mL certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2. Unpreserved samples may be sent to the laboratory within 2 weeks, then acid preserved and held 16 hours before analysis.	Fill to neck of bottle.	28 days	EPA 245; SM3112 (CVAA methods)
Metals, Primary (As, Sb, Ba, Be, Cd, Cr, Ni, Tl, Se)	Drinking Water	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2. Unpreserved samples may be sent to the laboratory within 2 weeks, then acid preserved and held 16 hours before analysis.	Fill to neck of bottle.	6 months	EPA 200
Metals, Secondary (Al, Fe, Mn, Ag, Zn)	Drinking Water	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2. Unpreserved samples may be sent to the laboratory within 2 weeks, then acid preserved and held 16 hours before analysis.	Fill to neck of bottle.	6 months	EPA 200; SM3120

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Metals, TCLP extract *(Note 7)*	Solid Waste	Three 1-L wide mouth glass bottles	Cool to ≤ 6°C. HNO_3 to pH < 2.	Fill to neck of bottle.	Metals: 6 months from field collection to TCLP extraction; 6 months from TCLP extraction to analysis: Mercury: 28 days from field collection to TCLP extraction; 28 days to analysis	SW-846 Method 1311, 6010, 6020
Metals, Total	Storm water, Surface water, and Wastewater	1-L certified metal-free glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	EPA 200; SM3111; SM3113; SM3120
Metals, Total	Sediment, Soils and Sludges	250-mL wide mouth glass or polyethylene jar	None required.	Fill to neck of bottle.	6 months	SW-846 Methods 6010, 6020
Metals, Total	Groundwater, Other aqueous waste	1-L glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SW-846 Methods 6010, 6020
Nitrate	Drinking Water	100-mL glass or polyethylene	Cool to ≤6°C.	Fill to neck of bottle.	48 hours	EPA 300; SM4110, SM4500-NO_3^-
Nitrate	Wastewater, Surface water, Storm water	100-mL glass or polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	EPA 300; EPA 352; SM4110; SM4500-NO_3^-
Nitrate	Groundwater, Other aqueous waste	1-L Teflon®, plastic, glass	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	SW-846 Method 6500, 9056, 9210
Nitrite	Drinking Water	100-mL glass or polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	EPA 300; SM4110, 4500-NO_2^-; SM4500-NO_3^-

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
Nitrite	Wastewater	100-mL glass or polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	EPA 300; SM4110, SM4500-NO_2^-; SM4500-NO_3^-
Nitrite	Groundwater, Other aqueous waste	100-mL glass or polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	SW-846 Method 9056, 9216
Nitroaromatics and Nitrosamines (Explosives)	Wastewater	1-L amber or foil wrapped glass bottles with TFE lined cap	Cool to ≤ 6°C; if residual chlorine present add 0.008% $Na_2S_2O_3$. Store in dark	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	EPA 625, SM6410
Nitroaromatics and Nitrosamines (Explosives)	Groundwater	4 x 1-L amber glass bottles with Teflon lined screw cap	Cool to ≤ 6°C; if residual chlorine is present, add 0.008% $Na_2S_2O_3$. Store in dark.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SW-846 Methods 8270, 8330, 8091
Nitroaromatics and Nitrosamines (Explosives)	Concentrated Waste Samples, Soils, and Sediments	250-mL amber wide mouth glass with Teflon®-lined lid	Store in the dark at ≤6°C.	Fill to neck of bottle.	Extract within 14 days; analyze within 40 days of extraction	SW-846 Methods 8270, 8330, 8091
Oil and Grease as Hexane Extractable Material	Soils and Sludges	250-mL wide mouth glass container fitted with a TFE-lined screw cap	HCl to pH < 2 for samples with aqueous layers. Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SW-846 Method 9071
Oil and Grease as Hexane Extractable Material	Surface water, Storm water, Wastewater	1-L wide mouth glass container fitted with a Teflon®-lined cap	H_2SO_4 or HCl to pH < 2. Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SM5520, EPA 1664
Orthophosphate	Drinking Water	100-mL glass or polyethylene bottles	H_2SO_4 to pH < 2. Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA Method 365

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Orthophosphate	Drinking Water	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	Filter within 15 minutes. 48 hours	EPA Method 300; SM4110; SM4500-P
Orthophosphate	Surface water, Wastewater	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	Filter within 15 minutes. 48 hours	EPA 365, EPA 300; SM4110; SM4500-P
PCBs	Drinking Water	4 x 1-L amber glass bottles with Teflon®-lined lids	If chlorine is present, first add 50 mg/L of sodium sulfite. Adjust the sample to pH < 2 by adding 6N HCl. Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 1 year; analyze within 1 year of extraction	EPA 508, 525
PCBs	Groundwater, Other aqueous waste	4 x 1-L amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	Extract within 1 year; analyze within 1 year of extraction	SW-846 Method 8082
PCBs	Sediment, Soils and Sludges, and other non-aqueous	250-mL wide-mouth glass container with Teflon®-lined lid	Cool to ≤ 6°C. Keep away from light.	Fill to neck of bottle.	Extract within 1 year; analyze within 1 year of extraction	SW-846 Method 8082
PCBs	Wipe Samples	250-mL wide-mouth glass container with Teflon®-lined lid	Cool to ≤ 6°C.	One wipe per jar.	Not established	SW-846 Method 3500, 3540, 3550, 8082
PCBs	Wastewater	1-L amber glass bottles fitted with Teflon®-lined screw cap	Cool to ≤ 6°C, if residual chlorine present add 0.008% $Na_2S_2O_3$.	Fill to neck of bottle.	Extract within 1 year; analyze within 1 year of extraction	EPA 608, 625, SM6410

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
PCBs (screening)	Drinking Water	2 x 1-L amber glass bottles	$HgCl_2$ to 10 mg/L. If chlorine is present, add 0.008% $Na_2S_2O_3$. Seal bottle and shake vigorously for 1 minute. Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 1year; analyze within 1 year0 days of extraction	EPA 508
Pesticides (Organochloral)	Drinking Water	2x 1-L amber glass bottles	Residual chlorine in the sample should be reduced by adding 50 mg/L of sodium sulfite. Adjust the sample to pH < 2 by adding 6N HCl. Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 14 days; analyze within 40 days of extraction	EPA 508
Pesticides (Organochloral)	Groundwater	Four 1-L amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SW-846 Method 8081, 8085
Pesticides (Organochloral)	Sediment, Soils and Sludges, Other Non-Aqueous	250-mL wide mouth glass container with Teflon®-lined lid	Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 14 days; analyze within 40 days of extraction	SW-846 Method 8081, 8085
Pesticides (Organochloral)	Wastewater, Surface water, Storm water	1-L glass bottles with Teflon®-lined cap	NaOH or H_2SO_4 to pH 5-9; Cool to ≤ 6°C *(Note 5)*.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SM6630; EPA 608

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Pesticides (Organophosphorus)	Drinking Water	1-L glass containers	If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Seal bottle and shake for 1 min. Cool to ≤ 6°C. Keep away from light.	Fill to neck of bottle.	Extract within 14 days; analyze within 14 days of extraction	EPA 507
Pesticides (Organophosphorus)	Groundwater	Four 1-L amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SW-846 Method 8085, 8141
Pesticides, TCLP extract *(Note 7)*	Solid Waste	Three 1-L wide mouth amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	14 days from field collection to TCLP extraction; 7 days from TCLP to preparative extraction; analyze within 40 days of preparative extraction	SW-846 Method 1311, 8141, 8085
pH	Drinking Water	Not applicable - field measurements	None required.	None.	Analyze in field	EPA 150, SM4500-H^+
pH	Groundwater, Other aqueous waste	50-mL glass or polyethylene bottles	None required.	None.	Analyze immediately	SW-846 Method 9040C, 9045
pH	Soils and Sludges	250-mL amber wide mouth glass jar or wide mouth polyethylene bottles	None required.	None.	Analyze immediately	SW-846 Method 9040, 9045
pH	Storm water, Surface water, Wastewater	50-mL glass or polyethylene bottles	None required.	None.	Analyze within 15 minutes	EPA 150; SM4500-H^+

Parameter	**Matrix**	**Sample Containers *(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method *(Note 3)***
Phosphorus	Surface water, Storm water	100-mL glass or polyethylene bottles	H_2SO_4 to pH < 2. Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA 365; SM4500-P
Polynuclear Aromatic Hydrocarbons (PAHs)	Groundwater	4 x 1-L amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SW-846 Method 8100, 8270, 8410
Polynuclear Aromatic Hydrocarbons (PAHs)	Wastewater	4 x 1-L glass bottles, amber with TFE-lined cap	If residual chlorine is present, add 0.008% $Na_2S_2O_3$. Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	EPA 610, 625, 1625; SM6410, 6440
Radiological Tests (alpha, beta, radium)	Groundwater	1-L glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SW-846 Methods 9310, 9315, 9320
Radiological Tests (alpha, beta, radium)	Drinking Water	1-L glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	EPA 900 (gross alpha, beta); EPA 903 (radium-226), EPA 904 (radium-228)
Radiological Tests (alpha, beta, radium)	Drinking Water	1-L glass or polyethylene bottles	HNO_3 to pH < 2.	Fill to neck of bottle.	6 months	SM7110 (alpha, beta), C (gross alpha); SM7500-Ra B, C (radium-226), D (radium-228)
Reactivity (releasable cyanide) *(Note 6)*	Solid Waste	Two 250-mL amber wide mouth glass jars or wide mouth polyethylene bottles	50% NaOH to pH >12. Store at ≤ 6°C.	Fill with no headspace and per sampling plan.	Analyze as soon as possible	SW-846 Method 9010, 9012, 9013, 9014 for cyanide

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Reactivity (releasable sulfide) *(Note 6)*	Solid Waste	Two 250-mL amber wide mouth glass jars or wide mouth polyethylene bottles	Fill the surface of the solid with 2N zinc acetate until moistened. Store at ≤ 6°C.	Fill with no headspace and per sampling plan.	Analyze as soon as possible	SW-846 Method 9030, 9031, 9215 for sulfides
Salinity	Surface water	Not applicable - field measurements	None required.	None.	Analyze in field	EPA 430/9-86-004
Semivolatile Organics	Drinking Water	Two 1-L amber glass bottles	Residual chlorine should be reduced at the sampling site by addition of 40-50 mg of sodium sulfite. The sample pH is adjusted to < 2 with 6N HCl. Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 14 days; analyze within 30 days of extraction	EPA 525
Semivolatile Organics	Groundwater, Other aqueous waste	Four 1-L amber glass bottles sealed vials equipped with unpierced Teflon®-lined septa	Cool to ≤ 6°C. Keep away from light. If chlorine is present, add 3mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	SW-846 Method 8270
Semivolatile Organics	Soils and Sludges, Sediment, and Other Non-Aqueous	250-mL wide mouth glass container with Teflon® lid	Cool to ≤ 6°C.	Fill to neck of bottle.	Extract within 14 days; analyze within 40 days of extraction	SW-846 Method 8270
Semivolatile Organics	Storm water	1-L amber glass bottles with Teflon®-lined caps	If chlorine is present, first add 0.008% $Na_2S_2O_3$. Cool to ≤ 6°C and keep in the dark.	Fill to neck of bottle.	Extract within 7 days; analyze within 40 days of extraction	EPA 625, 1625; SM6410
Semivolatile Organics	Concentrated Waste Samples	125-mL wide mouth glass container with Teflon® lid	None required.	Fill to neck of bottle.	Extract within 14 days; analyze within 40 days of extraction	SW-846 Method 8270

Parameter	Matrix	Sample Containers *(Note 2)*	Preservation	Filling Instructions	Holding Time	Method *(Note 3)*
Semivolatile Organics, TCLP extract *(Note 7)*	Solid Waste	Three 1-L wide mouth amber glass bottles with Teflon®-lined lids	Cool to ≤ 6°C. Keep away from light.	Fill to neck of bottle.	14 days from field collection to TCLP extraction; 7 days from TCLP to preparative extraction; analyze within 40 days of preparative extraction.	SW-846 Method 1311, 8270
Silica	Drinking Water	200-mL polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SM3120, SM4500-Si, SM4500 SiO_2
Silica	Wastewater	200-mL polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SM3120, SM4500-Si, SM4500 SiO_2
Sulfate	Drinking Water	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA 300, 375; SM4110, SM4500-SO_4^{2-}
Sulfate	Aqueous Waste	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	SW-846 Method 6500, 9035, 9036, 9038, 9056
Sulfate	Wastewater	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA 300, EPA 375; SM4110, SM4500-SO_4^{2-}
Sulfide	Drinking Water	500-mL glass or polyethylene bottles	None specified.	Fill to neck of bottle with minimal aeration.	Immediately	EPA 376
Sulfide	Solid Waste	500-mL glass or polyethylene bottles	Fill the surface of the solid with 2N zinc acetate until moistened. Store at ≤ 6°C.	Fill to neck of bottle with minimal aeration	7 days	SW-846 Method 9030, 9031, 9034, 9215

Parameter	**Matrix**	**Sample Containers** ***(Note 2)***	**Preservation**	**Filling Instructions**	**Holding Time**	**Method** ***(Note 3)***
Sulfide	Aqueous Waste	500-mL glass or polyethylene bottles	Use four drops of 2N zinc acetate solution per 100-mL of sample. Adjust the pH to greater than 9 with 6N NaOH solution. Store headspace free at ≤ 6°C.	Fill to neck of bottle with minimal aeration.	7 days	SW-846 Method 9030, 9031, 9034, 9215
Sulfide	Wastewater	100-mL glass or polyethylene bottles	Add 4 drops of 2N zinc acetate per 100-mL of sample; Add NaOH until pH > 9. Store at ≤ 6°C.	Fill to neck of bottle with minimal aeration.	7 days	SM4500 S^{2-}
Sulfite	Drinking Water	500-mL glass bottles	Sulfide may be removed by adding 0.5 g of zinc acetate. See method for additional steps to take for interferences.	Fill to neck of bottle.	Not specified	EPA 377
Sulfite	Wastewater	100-mL glass or polyethylene bottles	Analyze immediately or 1mL EDTA and cool to < 50°C.	Fill to neck of bottle.	Immediately (within 15 minutes)	SM4500-SO_3^{2-}
Temperature	Drinking Water, Storm water, Wastewater, Groundwater	Not applicable - field measurements	None required.	None.	Analyze immediately	SM2550
Total Kjeldahl Nitrogen	Surface water	500-mL glass or polyethylene bottles	H_2SO_4 to pH < 2; Cool to ≤ 6°C.	Fill to neck of bottle.	28 days	EPA 351; SM4500 NH_3, SM4500-N org
Total Organic Carbon (TOC)	Groundwater	1-L glass bottles with TFE-backed septa	H_2SO_4 to pH < 2; Cool to ≤ 6°C. Protect from sunlight.	Fill to neck of bottle.	28 days	SW-846 Method 9060

Parameter	Matrix	Sample Containers *(Note 2)*	Preservation	Filling Instructions	Holding Time	Method *(Note 3)*
TOC	Wastewater	100-mL glass bottles with TFE-backed septa	H_2SO_4 to pH < 2; Cool to ≤ 6°C. Protect from sunlight.	Fill to neck of bottle.	28 days	SM5310
Total Organic Halides (TOX)	Groundwater	250-mL amber Teflon Lined wide mouth glass jar	H_2SO_4 to pH < 2; Cool to ≤ 6°C.	Fill completely, no headspace.	14 days	SW-846 Method 9020
TOX	Wastewater	Amber glass with Teflon-lined caps	H_2SO_4 to pH < 2; Cool to ≤ 6°C.	Fill completely, no headspace.	14 days	EPA 450; SM5320
Total Petroleum Hydrocarbons (TPH)	Soils and Sludges	250-mL wide mouth glass container	Cool to ≤ 6°C.	Fill completely, no headspace.	Per sampling plan and state or local requirements	SW-846 Methods 3560, 8440, 4030, and 9074
Total Petroleum Hydrocarbons as Diesel Range Organics (DRO)	Soils and Sludges	250-mL wide mouth glass container	Cool to ≤ 6°C. See method for specific preservation criteria.	Fill completely, no headspace.	Extract within 14 days; analyze within 40 days of extraction	SW-846, Method 8015
Total Petroleum Hydrocarbons as Gasoline Range Organics (GRO)	Soils and Sludges	250-mL wide mouth glass container	Cool to ≤ 6°C. See method for specific preservation criteria.	Fill completely, no headspace.	14 days	SW-846, Method 8015
Total Solids	Surface water, Wastewater	100-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	7 days	SM2540
Total Suspended Solids	Storm water, Surface water, Wastewater	200-mL glass or polyethylene bottles	Cool to ≤ 6°C.	Fill to neck of bottle.	7 days	SM2540
Trihalomethanes (see DPBs)						
Turbidity	Drinking Water	1-L glass or polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	EPA 180; SM2130
Turbidity	Wastewater	100-mL glass or polyethylene	Cool to ≤ 6°C.	Fill to neck of bottle.	48 hours	EPA 180; SM2130

Parameter	Matrix	Sample Containers *(Note 2)*	Preservation	Filling Instructions	Holding Time	Method *(Note 3)*
Volatile Organic Compounds (VOCs)	Drinking Water	40-120-mL screw cap vials equipped with a Teflon® faced silicone septum–collect duplicates *(Note 4)*	HCl to pH < 2. Cool to ≤ 6°C. If chlorine is present, add 25mg ascorbic acid prior to HCl addition.	Fill completely, no headspace.	14 days	EPA 502 (GC), 524 (GC/MS), For specific volatile compounds review method requirements. By GC
VOCs	Groundwater	2 x 40-mL vials with Teflon® septum lined caps *(Note 4)*	HCl to pH < 2. Cool to ≤ 6°C. If chlorine is present, collect samples in 125-mL container with 4 drops of 10% $Na_2S_2O_3$ solution prior to HCl addition.	Fill completely, no headspace.	14 days	SW-846 Method 8260
VOCs	Wastewater and Storm water	40-mL glass VOA vial sealed with a septum *(Note 4)*	HCl or H_2SO_4 to pH < 2. Cool to ≤ 6°C. Presence of chlorine requires proper addition of $Na_2S_2O_3$ prior to adding acid.	Fill completely, no headspace.	14 days	EPA 624, 1624, 601; SM6200; SM6210, SM6230
VOCs	Concentrated Waste Samples	125-mL wide mouth glass container sealed with a septum *(Note 4)*	Cool to ≤ 6°C.	Fill completely, no headspace.	14 days	SW-846 Method 8260
VOCs, TCLP extract *(Note 7)*	Solid Waste	Two 40-mL glass vials *(Note 4)*	Cool to ≤ 6°C; adjust pH to < 2.	Fill completely, no headspace.	14 days from field collection to TCLP extraction; 14 days from TCLP extraction to analysis	SW-846 Method 1311, 8260

Notes:

Note 1. Check with the lab for container and preservative requirements and analytical method(s) to be used prior to sampling. Local or state regulations may supersede these requirements. In addition, sampling interferences, laboratory methods, and known matrix effects may require specific project preservations to be developed. A sampling plan is to be prepared and reviewed with the sampling laboratory and, in some cases, the regulator prior to starting any sampling/testing operation.

Note 2. Additional sample quantities may need to be collected in order to allow for the preparation and analysis of QC samples, such as MS and duplicates.

Note 3. Methods are listed without revision numbers or letters. Test procedures are periodically revised. Check applicable regulations (e.g., 40 CFR Part 136 for Wastewater; Parts 141 and 143 for Drinking Water; Parts 122–270 for Wastes under RCRA, etc.) for updates. State or local regulations should also be reviewed for requirements.

Note 4. VOA vials are to have Teflon®-faced silicone septum caps. 125-mL wide mouth glass containers may be used for high concentration solid and semi-solid samples only. All other jars and bottles are to have Teflon lined caps.

Note 5. Wastewater Pesticides: When single chemical category analytes are to be measured, the table should be followed. When two or more chemical categories are to be measured, cool the sample to ≤6°C, reduce chlorine residual with 0.008% sodium thiosulfate, store in dark, adjust the pH to 6–9, and hold for 7 days before extraction and 40 days after extraction. Only use sodium thiosulfate when chlorine is present. When measuring for benzidine, if 1,2-diphenylhydrazine is suspected, adjust pH to 4.0 +/- 0.2. The pH adjustment may be performed upon receipt at the lab or may be omitted if samples are extracted within 72 hours of collection. For analysis of aldrin, add 0.008% sodium thiosulfate ($Na_2S_2O_3$).

Note 6. Reactivity: Total analysis for cyanide and sulfide may be substituted if allowed in sampling plan. Preservation for total cyanide and sulfide is per Groundwater, Other aqueous waste, Soils, Sludges and Solid Waste found above.

Note 7. TCLP extraction per SW-846 Method 1311; extraction and analysis per determinative method.

Appendix C. Requirements for Collection of Quality Control Samples

Table C-1 specifies the recommended frequency for submitting quality control samples to the laboratory for analysis. The exact number and types of quality control samples is dependent on permit, regulatory and project data requirements. Except for sample containers submitted for MS/MSD lab studies, the laboratory should not be informed which samples are for QC purposes.

Test Samples. Samples collected and analyzed to provide data on the presence of chemicals within a specified sample matrix. A single test sample may require that a number of sample containers be prepared as described in Appendix B.

Field Duplicates. Submitted and analyzed to determine the repeatability of data. Generally, one field duplicate is submitted for each 10 test samples or another frequency based on project statistical design.

Trip Blanks. Trip blanks are VOA vials of ultra-pure water that are prepared and furnished by the laboratory. In general, one trip blank consisting of two VOA vials is included in each shipping container used to ship VOA water samples to check on possible sample contamination during shipment.

Equipment Decontamination Blanks. Submitted to check on the adequacy of field measures used to decontaminate sampling equipment. In general, one equipment decontamination blank is submitted for each type of sampling equipment. In compliance cases, this blank may be collected after field decontamination is performed. This process validates the equipment is cleaned properly between sampling locations.

Field Blanks. Submitted to check on the cleanliness of sample bottles and reagents used to prepare and preserve samples in the field. For volatiles, one field blank is submitted for each day of sampling or one per cooler. In other nonvolatile analytical procedures field blanks may be collected per batch of sample containers and preservatives used.

MS/MSD Samples. MS/MSD samples are prepared and run by the laboratory to verify the effectiveness of sample preparation procedures in measuring the chemicals of interest from the matrix material. In general, one MS/MSD sample is prepared and run by the laboratory for each type of analysis and matrix at a rate of one MS/MSD pair per 20 test samples (minimum one pair per batch of samples). The required frequency of MS/MSD analyses should be specified in the SAP and requested to be the specific matrix sampled.

Water Samples. Additional sample containers of water must be submitted to the lab to provide sufficient sample material for these tests.

Other Sample Material. Generally, the test sample provides sufficient material for the preparation of MS/MSD samples, and no additional sample containers are required by the lab.

Table C-1. Requirements for Quality Control Samples

Drinking Water (SDWA)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontamination Blanks**	**Field Blanks**	**MS/MSD Samples**
Residual chlorine, pH, temperature	N.A. – Field test				
Volatile Organic Compounds (VOCs)	None required *(Note 1)*	1 Trip Blank sample consisting of 2 lab filled VOA vials in each shipping container used to ship VOA water samples	*(Note 8)*	1 Field Blank sample consisting of 2 lab filled VOA vials exposed to the atmosphere where samples are collected. *Note 9* may be performed based on data needs	*(Note 4)*
Synthetic Organic Chemicals (Pesticides, Herbicides, PCBs)	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 4)*
Metals (Sb, As, Ba, Be, Cd, Hg, Cr, Ni, Tl, Se)	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 5)*
Lead and copper	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 5)*
Fluoride	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 5)*
Silica	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 5)*
Orthophosphate	None	None	*(Note 8)*	*Note 9* may be	*(Note 5)*

Drinking Water (SDWA)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontam-ination Blanks**	**Field Blanks**	**MS/MSD Samples**
	required *(Note 1)*			performed based on data needs	
Alkalinity	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None
Hardness	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None
Conductivity	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None
Turbidity	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None
Asbestos	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None
Total Coliform	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None
Nitrate	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 5)*
Nitrite	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	*(Note 5)*
Radionuclides	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None

Table C-1. Requirements for Quality Control Samples *(continued)*

Wastewater NPDES Permit Sampling (CWA) (Check state specific program for QC criteria)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontamination Blanks**	**Field Blanks**	**MS/MSD Samples**
Dissolved oxygen	N.A. – Field Test				
Biochemical Oxygen Demand (BOD)	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.
Chemical Oxygen Demand (COD)	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.
Total Organic Carbon (TOC)	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.
Oil and grease	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.
Metals	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.

Table C-1. Requirements for Quality Control Samples *(continued)*

Wastewater NPDES Permit Sampling (CWA) (Check state specific program for QC criteria)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontam-ination Blanks**	**Field Blanks**	**MS/MSD Samples**
Chromium, hexavalent (Cr^{+6})	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.
Pesticides	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.
PCBs	None required *(Note 1)*	None	None *(Note 8)*	*Note 9* may be performed based on data needs	None unless permit specified. One time demonstration of method performance per matrix.

Table C-1. Requirements for Quality Control Samples (continued)

Groundwater (RCRA; SW-846, July, 1992, Rev 1)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontamination Blanks**	**Field Blanks**	**MS/MSD Samples**
pH, temperature, conductivity	N.A. – Field test				
Volatile Organics	1 per day per matrix type *(Note 1)*	1 Trip Blank sample consisting of 2 lab filled VOA vials in each shipping container used to ship VOA water samples	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Semivolatile Organics	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Pesticides/PCBs/Herbicides	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Dioxins and Furans	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Metals	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 5)*
Cyanide	1 per day per matrix type *(Note 1)*	None	None	*(Note 9)*	1 in 20 *(Note 5)*
Total Organic Halides (TOX)	1 per day per matrix type *(Note 1)*	None	None	*(Note 9)*	1 in 20 *(Note 5)*
Radiological Tests	1 per day per matrix type *(Note 1)*	None	None	*(Note 9)*	1 in 20 *(Note 5)*

Table C-1. Requirements for Quality Control Samples (continued)

Other Waters And Non-Aqueous Liquids (RCRA; SW-846, July 1992, Rev 1)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontamination Blanks**	**Field Blanks**	**MS/MSD Samples**
Semivolatile Organics	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Pesticides/PCBs/Herbicides	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Volatiles Organics	1 per day per matrix type *(Note 1)*	1 Trip Blank Sample consisting of 2 lab filled VOA vials in each shipping container used to ship VOA water samples	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 4)*
Metals, Total	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 5)*
Cyanide	1 per day per matrix type *(Note 1)*	None	None	*(Note 9)*	1 in 20 *(Note 5)*
Mercury	1 per day per matrix type *(Note 1)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 5)*

Table C-1. Requirements for Quality Control Samples (continued)

Soils, Sludges, and Solid Waste (RCRA; SW-846, July 1992, Rev 1)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontamination Blanks**	**Field Blanks**	**MS/MSD Samples**
Semivolatile Organics, Pesticides/PCBs/Herbicides	1 per day per matrix type *(Note 2)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 6)*
Volatile Organics	1 per day per matrix type *(Note 2)*	1 Trip Blank Sample consisting of 1 lab filled clean material per VOA vial in each shipping container used to ship VOA samples	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 6)*
Metals and cyanide	1 per day per matrix type *(Note 2)*	None	1 per day per matrix type *(Note 7)*	*(Note 9)*	1 in 20 *(Note 6)*
PCB Wipe Samples (TSCA)					
Parameters	**Field Duplicates**	**Trip Blanks**	**Equipment Decontamination Blanks**	**Field Blanks**	**MS/MSD Samples**
PCBs	Based on sampling design *(Note 3)*	None	1 in 20; minimum one per day.	One per lot of wipes and solvent. *(Note 9)*	1 per lab batch of samples: submit 2 sample containers, each containing an unused wipe sample pad. Specify method for preparing MS/MSD.

Notes:

Field Duplicates

Note 1 Water samples:

For some projects and methods, 1 water sample in 10 may be required.

Prepare a Field Duplicate Sample, minimum one per day per matrix type for RCRA.

Note 2 Soil, sludge and solid waste:

For some methods and projects, 1 sample in 10 of the same matrix type (sand, silt, clay, etc.), may be required.

Prepare a Field Duplicate Sample, minimum one per day per matrix type for RCRA.

Note 3 PCB wipe samples:

For some methods and projects, 1 wipe sample in 10, may be collected as a duplicate wipe sample from an adjacent location on same type of surface;

Minimum one per day per type of surface and lot of wipe material (wood, metal, concrete, etc.).

MS/MSD

Note 4 Water samples for organic analyses:

Collect an additional four sample containers for 1 sample in 20 for each matrix type per method.

Note 5 Water samples for inorganic analysis:

One additional sample container may be required

Test sample should include enough material for lab MS/MSD samples to be run at the rate of 1 per 20 test samples

(Minimum of one MS/MSD per matrix per method or one per batch of samples).

Note 6 Soil, sludge, non-aqueous liquids, solid waste:

One additional sample container may be required

Test Sample may include enough material for lab MS/MSD samples to be run at the rate of 1 per 20 test samples

(Minimum of one MS/MSD per matrix per method or one per batch of samples).

Equipment Decontamination Blanks

Note 7 Submit one per type of analysis for each type of sampling equipment used for each day of sampling and each matrix sampled.

Project specific requirements may eliminate or increase the frequency of equipment decontamination blanks.

Note 8 Not required if sample containers are filled directly from the tap, outfall or are collected with pre-cleaned dedicated sampling equipment.

Field Blanks

Note 9 Submit one per type of analysis for each batch of sample containers and preservatives used.

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Acronyms

40 CFR	Title 40 of the Code of Federal Regulations

A

AA	Atomic Absorption
AFI	Air Force Instruction
AL	Action Level
ANSI	American National Standard Institute
AR	Army Regulation
AR	Arsenic Rule
ARCS	Assessment and Remediation of Contaminated Sediments
ASQ	American Society for Quality
ASTM	American Society for Testing and Materials

B

BAT	Best Available Technology
BCT	Best Conventional Pollution Control Technology
BMP	Best Management Practices
BOD	Biochemical Oxygen Demand
BPT	Best Practicable Control Technology Currently Available
BSAF	Biota-Sediment Accumulation Factor
BTEX	Benzene, Toluene, Ethylbenzene, and Xylene

C

C	Celsius
CAA	Clean Air Act
CAD	Computer Aided Design
CAS	Chemical Abstract Service
CCV	Continuing Calibration Verification
CEM	Continuous Emissions Monitoring
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations

CGP	Construction General Permit
COC	Chain-of-Custody
COD	Chemical Oxygen Demand
COLIWASA	Composite Liquid Waste Sampler
COPC	Contaminants of Potential Concern
COR	Contracting Officer's Representative
CPT	Cone Penetration Testing
CSM	Conceptual Site Model
CVAFS	Cold Vapor Atomic Fluorescence Spectrometry
CWA	Clean Water Act
CWS	Community Water System

D

DBP	Disinfection byproduct
DDBPR	Disinfectants and Disinfectant Byproducts Rule
DENIX	Defense Environmental Network and Information Exchange
DERP	Defense Environmental Restoration Program
DGT	Diffusion Gradients in Thin films
DHC	Dehalococcoides
DNAPL	Dense, Non-Aqueous Phase Liquid
DO	Dissolved Oxygen
DOC	Dissolved Organic Carbon
DoD	Department of Defense
DODI	DoD Instruction
DON	Department of the Navy
DOT	Department of Transportation
DPD	Diethylphenylenediamine
DPT	Direct Push Technology
DQO	Data Quality Objectives
DTIC	Defense Technical Information Center
DTPA	Diethylenetriaminepentaacetic Acid
DU	Decision Unit

E

EA	Environmental Assessment
EAB	Enhanced Anaerobic Bioremediation
ECD	Electron Capture Detector
ECE	Environmental Compliance Evaluation
EDD	Electronic Data Deliverable
EDTA	Ethylenediaminetetraacetic Acid
EIS	Environmental Impact Statement
ELAP	Environmental Laboratory Accreditation Program
EMR	Electromagnetic Radiation
EMS	Environmental Management System
ENVCAP	Environmental Compliance Assistance Platform
EOD	Explosive Ordnance Disposal
EPA	Environmental Protection Agency
EPCRA	Emergency Planning and Community Right-to-Know Act
EPTDS	Entry Point to the Distribution System
ESRI	Environmental Systems Research Institute
ESS	Explosives Safety Submission

F

FAA	Federal Aviation Administration
FAR	Federal Acquisitions Requirements
FAS	Ferrous Ammonium Sulfate
FFCA	Federal Facility Compliance Act
FID	Flame Ionization Detector
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FLB/FN	Field Log Book/Field Notes
FONSI	Finding of No Significant Impact
FSP	Field Sampling Plan
FWPCA	Federal Water Pollution Control Act

G

GAC	Granular Activated Carbon

GC	Gas Chromatography
GCP	General Construction Permits
GIS	Geographic Information System
GP	General Storm Water Permit
GPS	Global Positioning System
GWR	Ground Water Rule

H

HAA5	Haloacetic acids
HASP	Health and Safety Plan
HCL	Hydrochloric Acid
HDPE	High-Density Polyethylene
HIS	Hazard Information System
HMIS	Hazardous Materials Identification System
HSWA	Hazardous and Solid Waste Amendments

I

IATA	International Air Transport Association
IC	Ion Chromatograph
ICAO	International Civil Aviation Organization
ICP-AES	Inductively Coupled Plasma-atomic Emission Spectrometry
ICV	Initial Calibration Verification
IDLH	Immediately Dangerous to Life and Health
IDW	Investigation Derived Waste
IEC	International Electrotechnical Commission
IESWTR	Interim Enhanced Surface Water Treatment Rule
IMS	Immunomagnetic Separation
IR	Installation Restoration
ISM	Incremental Sampling Methodology
ISO	International Organization for Standardization
ITRC	Interstate Technology & Regulatory Council

K

KCL	Potassium Chloride

KI	Potassium Iodide
Kow	Octanol-Water Partition Coefficient

L

LCR	Lead and Copper Rule
LCS	Laboratory Control Sample
LNAPL	Light, Non-Aqueous Phase Liquid
LTTD	Low-Temperature Thermal Desorption
LUST	Leaking Underground Storage Tank

M

MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MCA	Multichannel Analyzer
MCL	Maximum Contaminant Level
MCLG	Maximum Contaminant Level Goal
MCO	Marine Corps Order
MCR	Manual Request Change
MDL	Method Detection Limit
METCAL	Metrology and Calibration
MGD	Million Gallons per Day
MIS	Multi-Increment Sampling
MNA	Monitored Natural Attenuation
MQO	Measurement Quality Objective
MS4	Municipal Separate Storm Sewer Systems
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
MSGP	Multi-Sector General Permit

N

$NaBH_4$	Sodium Borohydride
NAVFAC	Navy Facilities Engineering Command
NAVOSH	Navy Occupational Safety and Health
NCP	National Oil and Hazardous Substances Pollution Contingency Plan

NCWS	Non-Community Water System
NEHC	Navy Environmental Health Center
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NEPA	National Environmental Policy Act
NFPA	National Fire Protection Association
NIPDWR	National Interim Primary Drinking Water Regulations
NMCPHC	Navy & Marine Corps Public Health Center
NOAA	National Oceanographic and Atmospheric Administration
NOEC	No-effect Concentration
NOI	Notice of Intent
NOS	Not Otherwise Specified
NOSSA	Naval Ordnance Safety and Security Activity
NOT	Notice of Termination
NPDES	National Pollution Discharge Elimination System
NPDWR	National Primary Drinking Water Regulation
NRC	National Research Council
NSDWR	National Secondary Drinking Water Regulation
NTNCWS	Non-Transient Non-Community Water System
NTU	Nephelometric Turbidity Units

O

O&G	Oil and Grease
O&M	Operations and Maintenance
OD	Outside Diameter
OSHA	Occupational Safety and Health Administration
OSH Act	Occupational Safety and Health Act

P

PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbons
PAL	Project Action Level
PCB	Polychlorinated Biphenyl

PDB	Passive Diffusion Bag
PDMS	Poly(dimethylsiloxane)
PDS	Post-digestion Spikes
PE	Performance Evaluation
PE	Polyethylene
PEL	Permissible Exposure Limit
PID	Photoionization Detector
PISCES	Passive In situ Concentration Extraction Sampler
PM	Preventative Maintenance
PMN	Premanufacturing Notice
POC	Point of Contact
POCIS	Polar Organic Chemical Integrative Sampler
POM	Polyoxymethylene
POX	Purgeable Organic Halogen
POP	Proof of Performance
PPB	Parts per Billion
PPE	Personal Protective Equipment
PPM	Parts per Million
PQO	Project Quality Objective
PT	Proficiency Test(ing)
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl Chloride
PWS	Public Water System

Q

QA	Quality Assurance
QAM	Quality Assurance Manual
QAP	Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QC	Quality Control
qPCR	Polymerase Chain Reaction

R

RAO	Remedial Action Objective
RCRA	Resource Conservation and Recovery Act
Rdase	Reductive Dehalogenase
RESS	Required Explosives Safety Submission
RFA	RCRA Facility Assessment
RFI	RCRA Facility Investigation
RFQ	Requests for Quote
RI	Remedial Investigation
ROD	Record of Decision
RPD	Relative Percent Difference
RPPM	Respiratory Protection Program Manager
RQ	Reportable Quantities

S

SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act of 1986
SD	Serial Dilution
SDWA	Safe Drinking Water Act
SI	Site Inspection
SIC	Standard Industrial Classification
SLP	Slope
SOC	Synthetic Organic Chemical
SOH	Safety and Occupational Health
SOP	Standard Operating Procedure
SOW	Scope of Work
SPCC	Spill Prevention Control and Countermeasure
SPMD	Semipermeable Membrane Device
SPME	Semipermeable Microextraction Device
SPP	Systematic Planning Process
SQuiRT	Screening Quick Reference Table
S/S	Solidification/Stabilization
SVE	Soil Vapor Extraction
SVOC	Semivolatile Organic Chemical

SWDA	Solid Waste Disposal Act
SWPPP	Storm Water Pollution Prevention Plan
SWTR	Surface Water Treatment Rule

T

TCDD	Tetrachlorodibenzo-p-dioxin
TCDF	Tetrachlorodibenzofuran
TCE	Trichloroethylene
TCLP	Toxic Characteristic Leaching Procedure
TCR	Total Coliform Rule
TDS	Total Dissolved Solids
THM	Trihalomethane
TMDL	Total Maximum Daily Loads
TNCWS	Transient Non-Community Water System
TOC	Total Organic Carbon
TOX	Total Organic Halogen
TPH	Total Petroleum Hydrocarbons
TRI	Toxic Release Inventory
TSA	Technical Systems Audit
TSCA	Toxic Substances Control Act
TSDF	Treatment, Storage, and Disposal Facility
TSS	Total Suspended Solids
TTHM	Total Trihalomethane

U

UA	Urbanized Area
UCMR	Unregulated Contaminants Monitoring Rule
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
UIC	Underground Injection Control
USACE	United States Army Corps of Engineers
USGS	U.S. Geological Survey
UST	Underground Storage Tank

V

VOA	Volatile Organic Analysis
VOA	Volatile Organic Analytes
VOCs	Volatile Organic Chemicals
VSP	Visual Sample Plan

Glossary of Terms

A

Absorption	Assimilation of molecules or other substances into the physical structure of a gas, liquid, or solid without chemical reaction. The penetration of one substance into the inner structure of another.
Accuracy	The degree of agreement between a measured value and the true value. The combination of bias and precision of an analytical procedure, which reflects the closeness of a measured value to a true value.
Accreditation	A formal recognition that an organization (e.g., laboratory) is competent to carry out specific tasks (e.g., tests) or specific types of tasks.
Acetone	Volatile organic solvent, soluble in water. A listed hazardous waste in RCRA. Other names include: dimethylketone; 2-propanone
Acidity	The quantitative capacity of aqueous media to react with hydroxyl ions at a designated pH.
Action Level (AL)	Concentration that determines the need for evaluation of treatment requirements.
Active Sampler	In air monitoring context, a sampling device that uses a pump or vacuum to pull ambient air through the monitor or collection media.
Actual Lapse Rate	The rate of temperature change as an air mass rises or falls when factors of temperature gradients, humidity, etc. are taken into account.
Acute Exposure	An exposure to a toxic substance that results in severe biological harm or death in a short period of time.
Adsorption	Physical adhesion of molecules or colloids to the surfaces of solids without chemical reaction. One substance is attracted to and held on the surface of another. Adherence of the atoms, ions, or molecules of a gas or liquid to the surface of another substance.
Aerosol	Particulates of either liquid droplets or solids suspended in air. Aerosols most important in air pollution are in the 0.01–100 µm particle size range.
Amberlite®	Brand name for an XAD-2 resin; a polymeric material used for collection of polynuclear aromatic hydrocarbons from air.
Ambient	An encompassing state or naturally occurring environment.

Ambient Water	Waters in their natural state in the natural environment (e.g., rivers, lakes, streams, estuaries, and other receiving waters), as opposed to effluent discharges.
Analyte	The element, compound, or substance to be measured by chemical analysis.
Anemometer	An instrument used to measure wind speed.
Apparatus	The equipment used to collect samples, prepare samples, or measure analytes, such as: sample containers and other containers, filters, filter holders, labware, tubing, pipets, and other materials and devices that will contact samples, blanks, or analytical standards.
Aquifer	A layer of porous rock or soil that is saturated with water.
Artesian Well	A well made by boring into the earth until water is reached, and because of internal pressure, flows up like a fountain.
Artifacts	Substances produced during sampling or analysis that are not part of the desired sample. They may arise from equipment or environmental contamination, chemical interactions, or thermal effects.
Asbestos	Any material containing more than one (1) percent of mineral fibers from minerals such as chrysotile, amosite, crocidolite, or tremolite. A group of impure magnesium silicate minerals that occur in fibrous form.
Asphyxiant	A vapor or gas which can cause unconsciousness or death by suffocation (lack of oxygen). Simple asphyxiants are non-reactive gases such as nitrogen, carbon dioxide, and freon.
Atmospheric Stability	Refers to the vertical motions of the atmosphere that affect pollutant dispersion in the various atmospheric layers.
Atmospheric Turbulence	Swirls of motion of various air masses in the atmosphere (eddies or eddy currents).
Atomic Absorption (AA)	A technique for analyzing metals. The substance to be measured is converted to an atomic vapor by spraying a solution into a flame. An elemental light beam is directed through the flame, into a monochromator, and then to a detector that measures the amount of light absorbed by the atomized element in the flame. The absorbance at the selected wavelength is measured and compared with that of a reference substance. The absorbance is proportional to the concentration. This technique usually measures one element at a time.

B

Background Samples	Samples that define the concentration of various chemicals in normal uncontaminated site material. Background samples are used to compare site conditions to the surrounding environment.

	Background samples are collected and handled in the same manner as all other samples.
Bailer	A long, cylindrical sampling device lowered into a well on a rope or cable and filled with a ground water sample.
Biased Sample	Samples that are collected at locations that are chosen based on historical information, behavior of contaminants, and knowledge about the physical system's matrix or the physical system's effect on the contaminant's transport or fate.
Biocide	A preservative (usually acid, to lower the pH) added to the sample to prevent microbial degradation.
Biodegradation	The process of decaying caused by living organisms.
Biota	Living creatures such as plants, animals, insects, fungi, bacteria.
Blank (sample)	Sample that does not contain the analyte of interest but in other respects has, as far as possible, the same composition as the actual sample.
Blind Samples	A quality control sample in which the laboratory performing the analysis is unaware of the sample origin. This sample may be collected as a duplicate during field operations or may be prepared from a known reference material containing the constituent of interest.
Borosilicate	A type of acid-resistant glass.
Boundary Layer	Layer of atmosphere influenced by ground conditions.
Buddy System	System of organizing employees into work groups in such a manner that each employee of the work group is designated to be observed by at least one other employee in the work group. The purpose of the Buddy System is to provide rapid assistance to employees in the event of an emergency.
Buoyant Turbulence	In air monitoring context, the air circulation that results from warm air rising from the earth's surface and being replaced by cooler downward moving air.

C

° C	Degrees Centigrade (Celsius). Water freezes at 0° C and boils at 100° C at normal pressure.
Calibration	The set of operations, which establish, under specified conditions, the relationship between values indicated by a measuring instrument, measuring system, or values represented by a material measure, and the corresponding known values of a measure.
Carcinogen	A substance capable of causing cancer.

Certification	Procedure by which a regulatory agency or third party gives written assurance that a product, process, or service conforms to specified requirements.
Chain-of-Custody Record	A document or documents that record the time and date of persons responsible for the security of samples from the time the samples are collected until the samples are consumed in testing or discarded.
Chemical Derivatization	Technique used for analysis of specific pollutants or compounds by chemically reacting or modifying the compound with a chemical reagent (derivatization reagent) to form a compound that is detectable and readily identified.
Chlorofluorocarbons (CFCs)	A class of chemicals that contain both chlorine and fluorine. CFCs are used as refrigerants, cleaning solvents and aerosol propellants.
Chlorinated Solvents	Organic chemicals that contain chlorine and that are liquids at normal temperatures and pressures.
Chromatogram	A plot displaying peaks that correspond to the constituents detected by a gas, liquid, or ion chromatograph.
Chronic Effect	Adverse health effects resulting from repeated doses of, or exposures to, a substance over a relatively prolonged period of time.
Cleanup Standard	The level of contamination that is acceptable in materials that remain after the cleanup of a contaminated site.
Coliform Bacteria	Bacteria used as indicator of pollution if found in water. Gram-negative, non-spore forming rods that ferment lactose within 48 hours at 35° C.
Collector Efficiency	The degree to which a sampling device is capable of collecting the desired analyte(s) without physically or chemically altering the analyte(s).
Co-located Samples	Two or more portions collected at the same point in time and space so as to be considered identical.
Colorimetric	Describes any technique by which an unknown color is visually evaluated in terms of standard colors. The concentration of a compound can be measured quantitatively from the shade or intensity of the test color or the length of the test color stain in a scaled vessel.
Combustible Gas Indicator (CGI)	Instrument used to determine the potential for combustion or explosion in an unknown atmosphere.
Combustible Liquid Class II (OSHA)	Class II liquids include those with flashpoints at or above 100° F. (37.8° C.), and below 140° F. (60° C.) except a mixture having components with flashpoints of 200° F. (93.3 C.) or higher,

	the volume of which make up 99 percent or more of the total volume of the mixture (Title 49 CFR 176.30).
Combustible Liquid (DOT Usage)	Flashpoint 100° F – 200° F.
Community Water System (CWS)	A public water system that serves at least 15 service connections used by year-round residents or regularly services at least 25 year-round residents.
Comparability	Degree with which data from one study may be compared to other similar studies.
Completeness	Measure of the amount of collected data compared to the amount of data expected or required under ideal conditions.
Component Air Sample	An air sample taken using a collection device that favors collection of either a specific pollutant or a class of pollutants.
Composite Sample	A sample prepared by physically combining two or more samples having the same specific relationship and processed to ensure homogeneity. For example, flow proportioned, time proportioned or equal mass samples.
Compressed Gas	Material packaged in a cylinder, tank or aerosol under pressure exceeding 40 psi at 70° F or other pressure parameters identified by DOT.
Condensible	Capable of forming a liquid from a vapor.
Confidence Interval	Range of values that can be declared with a specified degree of confidence to contain the correct or true value.
Confined Space	Any space that has restricted access and is not normally occupied. Includes tanks, vaults, and holes more than 4 feet deep, etc.
Contamination	Something inadvertently added to the sample during the sampling and analytical processing resulting in positive or negative effects.
Contamination Reduction Zone	At a remediation site, the transition zone between contaminated area (Exclusion Zone) and clean area. The zone is where personnel remove their personal protective equipment.
Continuous Sampler	Sampler that withdraws a sample constantly from a stream and accumulates the withdrawn volume for analysis at a later time.
Control Charts	A graph of some measurement plotted over time or sequence of sampling, together with control limit(s) and, usually, a central line or warning limit(s). Control charts may include a means chart for standards, matrix spikes, calibration check standards, laboratory control standards, a means chart for background or blank results, and a range chart for replicate analysis.
Control Sample	The sample against which results are compared or judged. The sample is identical in matrix whenever possible and prepared under the same conditions to determine the extent of variations.

	Control samples may include blanks, standards, check samples, or split samples, etc., which establish intralaboratory or analyst specific precision and bias, and to assess the performance of all or a portion of the measurement system, including sampling.
Conventional Pollutants	In the National Pollution Discharge Elimination System (NPDES) program, EPA Wastewater Discharge Information, Application Form 2C, the pollutants found in Part A.1. These are Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Organic Carbon (TOC), Total Suspended Solids (TSS), Ammonia, pH, and Temperature.
Coroliolis Force	A fictitious force used mathematically to describe the motion, as of aircraft, cloud formations and air masses, relative to a non-inertial, uniformly rotating frame of reference such as the Earth.
Corrosive	DOT Definition: A liquid or solid, excluding poisons, that causes visible destruction or irreversible alterations in human skin tissue at the site of contact, or has a severe corrosion rate on steel. See Title 49 CFR 173.240. In chemical terms, any solid, liquid, or gaseous substance that attacks materials and metals, or burns, irritates, or destructively attacks organic tissue. See 40 CFR Part 264 for definition of corrosivity under RCRA.
Cryogen	A substance for obtaining low temperatures (refrigerant). Cryogenic liquids such as liquid nitrogen are hazardous to handle.
Cyclone Collector	An inertial, centrifugal collection device for solid particles that separates particles by whirling them in a cyclonic motion inside a conical shaped collector.

D

Data Quality	The totality of features and characteristics of data that bears on their ability to satisfy a given purpose; the sum of the degrees of excellence for factors related to data.
Data Quality Objectives (DQOs)	Qualitative and quantitative statements which specify the study objectives, domain, limitations, the most appropriate types of data to collect, and specifies the levels of decision error that will be acceptable for the project.
Decontamination	Removal of substances from skin, clothing, or equipment to the extent necessary to preclude the occurrence of foreseeable health effects, environmental effects, or sampling and testing effects.
Deionized Water	Water prepared by passing feedwater through a mixed-bed ion exchanger consisting of strong anion and strong cation resins mixed together. The resultant water has the characteristics of Type I or Type II water depending on the purification process. (See Distilled Water)

Denuders	Glass tubes (or sometimes glass tubes packed with glass beads) coated with an adsorbent chemical film for trapping specific gaseous analytes during air sampling.
Diethylphenylenediamine (DPD)	The reagent used in a colorimetric test to determine residual free chlorine (disinfectant) concentrations.
Direct Assessment of Air Pollution	Method of assessment where emission rates are measured directly rather than by determining downwind mass/volume concentrations.
Dispersion Modeling of Air Pollution	The use of meteorological, topographical, and specific pollutant concentration data to predict larger range effects of pollution. Method of iterative assessment using measurements made downwind of source combined with assessments of meteorological and topographical factors in a complex computer model.
Dissolved Matter	A substance that is dispersed in a material to give a single homogeneous phase.
Dissolved Oxygen	Oxygen dissolved in water.
Distilled Water	Water prepared by thermal distillation using a still of all-borosilicate glass, fused quartz, tin or titanium with the distillate meeting the following characteristics of Type I (Type II) water: Resistivity (megohm-cm @ 25° C) greater than 10 (> 1); Conductivity (µmhos/cm @ 25° C.) < 0.1 (= 1); Total oxidizable organic carbon (mg/liter) < 0.05 (< 0.2); Total solids (mg/liter) less than or equal to 0.1 (equal to 1); SiO2 (mg/liter) < 0.05 (< 0.1). These characteristics are from Standard Methods for the Examination of Water and Wastewater, 18th edition; other type waters may be specified in the work plan.
Dose	The amount of energy or substance absorbed in a unit volume of an organ or individual.
Dose Rate	The dose delivered per unit of time.
Dredge	A type of sampling equipment used to collect samples of sludge or sediments from the bottom of ponds, rivers, etc.
Duplicate (Sample)	Two samples taken from and representative of the same population and carried through all steps of the sampling and test procedure in an identical manner. Used to assess variance of the total method including sampling and analysis.
Dust	Solid particles generated by handling, crushing, grinding, rapid impact, detonation, and decrepitation of organic or inorganic materials, such as rock, ore, metal, coal, wood, and grain. Dusts do not tend to flocculate except under electrostatic forces; they do not diffuse in air but settle under the influence of gravity.

E

E. Coli	A member of the coliform group that can cause severe gastroenteritis, including diarrhea and vomiting (Escherichia coli).
Effluent	Liquid discharges from a piece of equipment or a site.
Effluent Limitations	Specific limits on the pollutants in the water discharged from a site.
Eijkelcamp Stony Soil Auger	A type of auger designed to make holes in stony soil.
Electrostatic Separator	An air sampler that uses high voltage to ionize aerosols then coalesce and collect them.
Entropy	A measure of a system's capacity to undergo change; a measure of the disorder in a system.
Entry Point	A place where water enters the distribution system of a drinking water supply system.
Environmental Samples	Samples of naturally occurring matrices such as soil, sediment, groundwater, surface water, and air.
Equipment Blank	An aliquot of reagent water that is subjected to all aspects of sample collection and analysis including contact with all sampling devices and apparatus. The purpose of the equipment blank is to determine if the sampling devices and apparatus for sample collection have been adequately cleaned.
Exclusion Zone	Designated zone at a hazardous waste site where contamination is known to or may occur. The exclusion zone may only be entered by trained personnel who are wearing appropriate personal protective equipment.
Exemption	A timely delay or release from compliance due to compelling factors (such as economics).
Explosimeter	Real time instrument that measures the concentration of organic chemicals in air as a fraction of the concentration required to cause an explosion
Explosion-proof Equipment	Apparatus enclosed in a case that is capable of withstanding an explosion of a specified gas or vapor and is capable of preventing the ignition of a specified gas or vapor surrounding the enclosure by sparks, flashes, or explosion of the gas or vapor within the case, and that operates at an external temperature such that a surrounding flammable atmosphere will not be ignited.
Explosive Division/Class 1.1	Mass-detonating material (high explosive).
Explosive Division/Class 1.2	Non-mass-detonating material; primarily fragmentation hazards.
Explosive Division/Class 1.3	Mass-deflagrating material (severe fire hazard).
Explosive Division/Class 1.4	Non-mass deflagrating material (fire hazard).

Explosive Limits	Some materials have a minimum and maximum concentration in air that can be detonated by spark, shock, fire, etc. The lowest concentration is known as the lower explosive limit (LEL). The highest concentration is known as the upper explosive limit (UEL).
Exposure	Subjection of a person to a toxic substance or harmful physical agent in the course of employment through any route of entry (e.g., inhalation, ingestion, skin contact, or absorption); includes past exposure and potential (e.g., accidental or possible) exposure, but does not include situations where the employer can demonstrate that the toxic substance or harmful physical agent is not used, handled, stored, generated, or present in the workplace in any manner different from typical non-occupational situations. An exposure to a substance or agent may or may not be an actual health hazard to the worker.
Extraction	Process where materials are separated using physical or chemical means.

F

° F	Degrees Fahrenheit. Water freezes at 32° F and boils at 212° F at normal pressure.
Fecal Coliforms	A group of coliform bacteria, associated with fecal matter of warm-blooded animals, whose presence in drinking water indicates the possible presence of disease-causing organisms and indicates the water is not safe for human consumption. Their confirmed presence is considered an acute violation of the Total Coliform Rule.
Field Blank	A quality control sample used to indicate potential sample contamination from sampling equipment or ambient air.
Field Duplicates (FD1 and FD2)	Two separate samples collected in separate sample bottles at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures, which may include filtration. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
Field Sampling Plan	A document that describes exactly where and how samples are to be collected, preserved, packaged, and shipped to the laboratory. The Field Sampling Plan also describes all required field tests and the chain of custody procedures.
Field Standards	Samples of known concentrations that are used in the field for calibration and comparison with unknown samples or to verify field sampling or test procedures.
Filter Pack	An air sampling device consisting of a series of two or more stages of filters for collection of specific analytes. The filters

	are separated by inert o-rings and the filter housing is also inert, usually Teflon.
Fixed Matter	Residues from the ignition of particulate or dissolved matter, or both.
Flame Ionization Detector (FID)	A detector that uses hydrogen flame to measure the presence of volatile chemicals in air. An air monitoring instrument that uses an FID detector in measuring health and safety exposure based on the total relative levels of volatile organics in the environment compared to background levels or a standard mixture.
Flammable (DOT Usage)	Flashpoint < 100° F.
Flammable Aerosols	An aerosol which is required to be labeled “Flammable” under the United States Federal Hazardous Substances Labeling Act. For storage purposes, flammable aerosols are treated as Class IA liquids (NEPA 30, Flammable and Combustible Liquids Code).
Flammable Gas	Any compressed or liquefied gas, except an aerosol, is flammable if either a mixture of 13 percent or less (by volume) with air forms a flammable mixture or the flammable range with air is wider than 12 percent regardless of the lower limit (at normal temperature and pressure). (International Civil Aviation Organization (ICAO) Technical Instructions).
Flammable Limits	Flammable liquids produce (by evaporation) a minimum and maximum concentration of flammable gases in air that will support combustion. The lowest concentration is known as the lower flammable limit (LFL). The highest concentration is known as the upper flammable limit (UFL).
Flammable Solid (DOT Usage)	Any solid material, other than one classed as an explosive, that under conditions normally incident to storage is liable to cause fire through friction or retained heat from manufacturing or processing; or that can be ignited readily, and when ignited burns so vigorously and persistently as to create a serious storage hazard. Flammable solids, excluding Dangerous When Wet, are further defined in Title 49 CFR 173.150.
Flash Point	The lowest temperature at which a liquid gives off enough vapor to form an ignitable mixture with air and produce a flame when a source of ignition is present.
Flow Rate	The rate at which a stream of material passes by a point or through an orifice (e.g., cubic feet per minute of air, gallons per minute of water.)
Fly Ash	Non-combustible aerosol mineral solids formed from incomplete oxidation of coal or fuel oil, generally < 200 µm diameter.
Follow-up Monitoring	Testing defined in the SDWA after a PWS installs treatment. Follow-up monitoring is required for lead, copper, and other water quality parameters.

Fugitive Emission	A source of air pollution other than stacks or vents (i.e., connectors, gaskets, valves, etc.).
Full Protective Clothing	Personal protective equipment that completely protects a worker's skin from possible contact with toxic or corrosive gases, liquids or solids. This level of PPE is used where high chemical gas, vapor, or fume concentrations in air may have a corrosive effect on exposed skin, and/or where the chemical in the air may be readily absorbed through the skin to produce toxic effects. These suits are impervious to chemicals, offer full body protection, and include self-contained breathing apparatus (SCBA).
Fully Encapsulating Suits	Full chemical protective suits that are impervious to chemicals, offer full body protection from chemicals and their vapors/ fumes, and are used with SCBA.
Fume	Fume aerosol particulates from condensation of distillation vapors, calcination, or chemical reactions.
Fungicide	A chemical that prevents the growth of fungus and which is approved for use by the U.S. EPA.

G

Gas	A state of matter in which the material has very low density and viscosity; can expand and contract greatly in response to changes in temperature and pressure; easily diffuses into other gases; readily; and uniformly distributes itself throughout any container. A gas can be changed to the liquid or solid state by the combined effect of increased pressure and/or decreased temperature.
Gas Chromatograph (GC)	An instrument where the components of a mix are separated from one another by volatilizing the sample into a carrier gas stream and over a bed of packing with a solid support or through a long narrow capillary. Different components move through the packing at different rates and are detected separately by an appropriate detector. The different detectors can measure ionization, thermal conductivity changes, density differences, or other characteristics of the separated components.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	A technique used in combination with a gas chromatograph in which a small amount of separated components from the GC travel into a mass spectrometer through a vacuum and are exposed to a beam of electrons which ionizes the molecules and the fragments of the molecules into characteristic ions. The ions are separated according to their mass using electromagnetic fields and then the characteristic ion patters are detected. A library of known identities or patterns is used to determine which fragments are present and identify the compound present in the sample.

Geostatistics	Statistical methods that are used to calculate the expected concentration of chemicals in soil between sampling points.
Giardia Lamblia	A parasitic protozoan species that infects the gastrointestinal tract.
Glass Globe Samplers	Evacuated glass bulbs used to collect whole air samples.
Grab Sample	A sample that is representative of one specific sample site location at a specific time. A single sample that is collected at one point in time and place.
Gradient Wind	Free blowing wind above the boundary layer of the atmosphere.
Graphic Furnace Atomic Absorption (GFAA)	A technique used for measuring metals. Graphite furnace atomic absorption (GFAA) or electrothermal atomic absorption spectroscopy is based on the same principle as atomic absorption. In GFAA, the flame is replaced with an electrically heated atomizer or graphite tube. A sample volume is dispensed in the tube and heated in three or more stages. The final stage heats the tube to atomize the element being determined. An elemental light beam is directed through the furnace, into a monochromator, and then to a detector that measures the amount of light absorbed by the atomized element. The absorbance at the selected wavelength is measured and compared with that of a reference substance. The absorbance is proportional to the concentration. This technique usually measures one element at a time.
Gram	A metric unit of weight equal to about 1/28 ounce. One cubic centimeter of pure water weighs one gram. There are 1,000 grams in a kilogram.
Groundwater	The portion of the water beneath the land surface that is within the zone of saturation (below the seasonally high water table) where all pore spaces of the geologic formation are filled with water.
Grouting	Filling a borehole or well with an impervious material such as bentonite clay or cement.

H

Handling Time	The time between collection of samples and their delivery to the laboratory. The maximum allowable handling time may be specified in the Field Sampling Plan.
Hardness	In water chemistry, hardness is a term originally referring to the soap-consuming power of water; current practice defines total hardness as the sum of the calcium and magnesium concentrations expressed as calcium carbonate in milligrams/liter; certain test techniques for hardness measure the polyvalent cations used to precipitate soap; as such it is also taken to include iron and manganese. “Permanent hardness” is the excess of hardness

	over alkalinity; “temporary hardness” is hardness equal to or less than the alkalinity. These are also referred to as “non-carbonate” or “carbonate” hardness, respectively.
Hazard Class	A category of hazard associated with a hazardous material or hazardous waste that has been determined to pose an unreasonable risk to health, safety, and property when transported (see Title 49 CFR 171.8). The hazard class is defined by the U.S. DOT and published in Title 49 CFR 172.101. The hazard classes used in the United States include Explosive (Division/Class 1.1, 1.3, 1.4); Flammable Liquid; Flammable Solid; Corrosive Material; Nonflammable Gas; -1, -B, -C, -D, and -E; Etiologic Agent; Irritation Material; Organic Peroxide; Combustible Liquid; Flammable Gas; and Blasting Agent.
Hazardous Air Pollutant	A pollutant to which no ambient air quality standard is applicable but that may cause or contribute to an increase in mortality or in serious illness. For example, asbestos, beryllium, and mercury have been declared hazardous air pollutants.
Hazardous Chemicals	Chemicals or materials used in the workplace that are regulated under the OSHA Hazard Communication Standard or the “right-to-know’’ regulations in Title 29 CFR 1910.1200.
Hazardous Material	In a broad sense, a hazardous material is any substance or mixture of substances that can produce adverse effects on the health and safety of a human being or the environment. Legal definitions are found in individual regulations.
Hazardous Substances	Chemicals, mixtures of chemicals, or materials subject to the regulations contained in Title 40 CFR 116. For transportation purposes, a material, and its mixtures or solutions, identified by the letter “E” in column 2 of the Hazardous Material Table included in Title 49 CFR 172.101 when offered for transportation in one package, or in one transport vehicle if not packaged, and when the quantity of the material therein equals or exceeds the reportable quantity (RQ). For details, refer to Title 49 CFR 171.8 and Title 49 CFR 172.101.
Hazardous Waste	Any material listed as such in Title 40 CFR 261, Subpart D, that possesses any of the hazard characteristics of corrosivity, ignitability, reactivity, or toxicity as defined in Title 40 CFR 261, Subpart C, or that is contaminated by or mixed with any of the previously mentioned materials (See Title 40 CFR 261.3).
Hazardous Waste Landfill	An excavated or engineered area on which hazardous waste is deposited and covered; proper protection of the environment from the material to be deposited in the landfill requires careful site selection, good design, proper operation, leachate collection, and treatment through final closure.
Hazardous Waste Manifest	

(EPA Usage)	The shipping document, originated and signed by the waste generator or authorized representative that contains the information required by Title 40 CFR 262, Subpart B.
Health and Safety Plan (HASP)	A document that describes the hazards that may be encountered while collecting specified samples and specifies the required personal protective equipment and procedures that will minimize the risks. The contents of a Health and Safety Plan required for work at a hazardous waste site are specified by OSHA in Title 29 CFR 1910.120.
Holding Time	The time between the collection of environmental samples and the extraction or analysis of the samples. Maximum allowable holding times are specified by regulations for many analytes within each regulatory program.
Homogeneity	The degree of uniformity of structure or composition. The term is often used to describe a mixture, solution, or environmental sample comprised of substances that are uniformly dispersed. When discussing a sample, the degree to which each portion of the sample is representative of the whole sample.
Homogenization	Process whereby a sample is mixed until a consistent physical appearance is achieved.
Hydrogeology	The presence and movement of underground water as a function of the soil and rock layers that are present.

I

Ignitability (EPA Usage)	A liquid with a flashpoint less than 140° F.
Imhoff Cone	A cone shaped graduated vessel used to measure the volume of solids that will settle out of a liquid.
Immiscible	Two liquids that are not capable of mixing or blending; not mutually soluble.
Impactor	An instrument for collecting samples of suspended particles (as dust in air) by directing a stream of the suspension onto a surface or into a liquid.
Impermeable	Media through which substances cannot be diffused at any measurable rate.
Impinger	An air sampling collection device that collects gas forcibly against a surface. Both wet and dry impingement processes may be used.
In Situ	Within place; commonly used to refer to the location of activities (e.g., in situ soil vapor measurements).
Indirect Assessment	Method of assessment using a variety of samples from different Air Pollution ambient locations relatively close to the source to estimate concentrations via dispersion modeling equations.
Inductively Coupled Plasma	

(ICP, ICAP)	A technique used for metal analysis based on the principle of atomic emission. The high temperature of the plasma excites atomic ionization which results in ionic emission spectra. The efficient excitation results in low detection limits and large dynamic ranges of four to six orders of magnitude for many elements. This technique is especially useful for refractory metals and significantly reducing chemical interferences associated with flame atomic absorption. Instruments are capable of performing multi-element analysis.
Inductively Coupled Plasma/ Mass Spectrometer (ICP/MS)	A technique in which part of the ionic cloud from an ICP torch is introduced into a vacuum and bombarded with electrons. The ions are separated according to their mass using electromagnetic separation and then the characteristic ion patters are detected by a mass spectrometer. It can determine which fragments are present and therefore the identity of the metal analytes.
Inert	Exhibits no chemical reactivity.
Infiltration	The flow of fluid into a substance through pores or small openings. The word is commonly used to denote the flow of water into soil.
Ingestion	The process of taking substances into the body through the mouth, as in food, drink, medicine, etc.
Inhalation	The breathing in of a substance in the form of gas, vapor, fume, mist, or dust.
Injection Well	A well into which gas, air, oil, water, or waste is pumped.
Inorganic Chemical	Generally considered to embrace all chemical substances except hydrocarbons and their derivatives. In some cases inorganic and organic chemicals may not be easily distinguished such as organometallics, organic cyanides, etc.
Integrated Sample	In air monitoring context, refers to a sample collected over time.
Internal Standard	A material present in or added to samples in known amounts to serve as a reference measurement. The standard added to a test portion of a sample in a known amount and carried through the entire procedure as a reference for calibration and controlling the precision and bias of the applied analytical method.
Inversion	In air monitoring context, a very thermally stable atmospheric condition where a positive thermal gradient exists and the upper air is warmer than the lower air.
Irritant	Any material, liquid, or solid substance, that upon contact with fire or when exposed to air gives off dangerous or intensely irritating fumes, such as tear gas, but not including Poison Division/Class 2.3 or 6.1 material. Materials named as irritants are presented in Title 49 CFR 173.38.

Isokinetic Sampling	Sampling in which the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sample point.

K

Kilogram	A metric unit of weight, about 2.2 pounds.
Kilopascal (kPa)	A unit of measure for vapor pressure.

L

Lab Pack	A hazardous waste disposal container holding many other small volume containers of compatible chemicals from laboratory operations, packed with an inert filler, for shipping to an approved disposal operation.
Laboratory Decontaminated	The cleaning of sampling equipment and bottles in a controlled setting such as a laboratory or the cleaning of equipment and materials used in testing.
Lagoon	An artificial pond used for the storage or disposal of liquids.
Lanyard	A cord, thong, rope, or strap used for fastening.
Leachate	Liquid that seeps from the ground. In the laboratory test methods, liquid that has been exposed to a contaminated solid material for a defined period of time.
Leaching	Action whereby soluble constituents migrate through permeable media due to the vertical percolation/infiltration of a liquid.
Lead Solder	A solder containing 0.2 percent or more lead.
Leak or Leaking	A flaw, crack, or hole permitting accidental escape of fluid. Any instance in which an article, container, or equipment has any hazardous material (e.g., PCB) on any part of its external surface or has released this substance to the surrounding environment resulting in an environmental threat.
Liter	A metric unit of volume somewhat more than a quart. A liter of water weighs 1 kilogram or about 2.2 pounds
Lithology	Presence and location of different types of soil, bedrock, and groundwater.
Lower Explosive Limit (LEL)	Minimum concentration of a combustible gas in air that will explode when ignited.
Lower Flammable Limit (LFL)	The concentration of a chemical in air over a liquid that has been heated to its flash point

M

Matrix	Refers to the mixture of biological, chemical, and physical materials found in various types of environmental samples such as freshwater, seawater, sediment, and animal or plant tissues.

The materials can interfere with the analyses and, if not compensated for, can lead to false and invalid data.

Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	A quality control sample prepared by adding a known amount of analyte to the matrix sample. A known amount of analyte added to a second or duplicate matrix sample is the MSD. The matrix sample, MS, and MSD data are used to evaluate the recovery of the analyte from the matrix (bias) and evaluate the reproducibility of the analyte in the MS/MSD (precision). All samples are prepared and analyzed to determine the effect of the matrix on the procedures used for processing.
Maximum Contaminant Level (MCL)	Enforceable standard that specifies how much of a contaminant can be present in drinking water.
Maximum Contaminant Level Goal (MCLG)	Nonenforceable goal that specifies the level of contaminant at which there is no health concern.
Mechanical Turbulence	In air monitoring context, the effects of air masses flowing over rough or irregular surface elements.
Meteorological Parameter	Weather conditions such as wind direction and speed, temperature, atmospheric stability, atmospheric pressure, humidity, and precipitation that are very critical to air sampling and monitoring activities.
Method	A sampling or measurement process that has been officially specified by an organization as meeting its data quality requirements.
Method Blank	A quality control sample or blank processed simultaneously with and under the same conditions as the samples including all steps of the test procedure. The method blank may be reagent water, clean sand, or pure material known to be free of the analyte of interest and with a similar matrix as the samples being tested.
Method Detection Limit (MDL)	The minimum concentration of an analyte in a specific matrix and with a specific method, is measured and reported with a 99 percent confidence that the concentration of the analyte is greater than zero.
Microorganism	An organism of microscopic or ultramicroscopic size.
Milligram	A metric unit of weight. There are 1,000 milligrams in one gram of a substance.
Milliliter	A metric unit of volume equal to one cubic centimeter. A milliliter of pure water weighs one gram at 4° C and 760 mm mercury pressure.

Minimum Level (ML)	The lowest level at which the entire analytical system gives a recognizable signal and an acceptable calibration point.
Mist	Aerosol particulates arising from vapor condensation, chemical reactions, or liquid atomization.
Mixing Layer	In air monitoring context, the top of a neutral or unstable surface air layer. For stable surface air layers such as inversions, the mixing layer is undefined.
Mottles	Areas of different color or texture. Term often used in soil descriptions.
	N
Nebulization/Reflux	A method of air sampling in which the air stream is passed through a nebulized mist of liquid which traps the pollutants of interest.
Nitrile	A type of synthetic rubber that is resistant to oil and is commonly used to make gloves used when handling hazardous chemicals.
Nephelometric Turbidity Unit (NTU)	A unit of measure for turbidity of water.
Nontransient Non-community Water System (NTNCWS)	A public water system that is not a community water system and that regularly serves at least 25 of the same persons over 6 months per year.
	O
Olfactory	Of, relating to, or connected with the sense of smell.
Opacity	Extent to which a material absorbs or diffuses light.
Oxidizer	A chemical other than a blasting agent or explosive as defined in Title 29 CFR 1910.109(a), which initiates or promotes combustion in other materials thereby causing fire either of itself or through the release of oxygen or other gases.
	P
Particulate Matter	Solid matter that is heterogeneously dispersed in water. In air monitoring, the particulate matter may be droplets of liquid or solid particles, suspended in air and referred to as aerosols.
Passivate	In air or gas monitoring, the chemicals of interest are allowed to diffuse through the membrane or plastic film and are absorbed on a solid absorbent material inside to trap the chemicals.
Passive Sampler	In air or gas monitoring, refers to a sampling device that allows ambient air conditions to diffuse the sample through the monitor or onto the adsorbent material.

PCB Transformer	Any transformer that contains a liquid in which PCBs are present at a concentration of 500 ppm PCB or greater. (Title 40 CFR 761.3).
Percolating	A liquid that is flowing down through a porous material such as soil.
Permissible Exposure Limit (PEL)	The maximum allowable 8-hour time-weighted average concentration of a chemical in air to which workers may be exposed.
Pesticide	A chemical that is toxic to pests (insects and rodents) and that is approved by EPA for use for this purpose.
pH	The negative logarithm of the hydrogen-ion activity in an aqueous solution or the logarithm of the reciprocal of the hydrogen-ion activity. pH is a measure of the acidity or alkalinity of a liquid on a scale from 1 to 14; acids have a pH lower than 7; alkalis have a pH above 7.
Phase I	The first phase of EPA’s regulatory development approach to control air pollution. It covers the first eight VOCs to be regulated.
Phase II	The second phase of EPA’s regulatory approach to control air pollution. Phase II covers additional VOCs, pesticides, and inorganic chemicals.
Phenolic Compounds	Aromatic organic chemicals that contain one or more hydroxyl groups bonded directly to the benzene ring.
Photo Ionization Detector (PID)	A detector that uses an ultraviolet light to measure the presence of volatile chemicals in air. An air monitoring instrument that uses the PID detector in measuring health and safety exposure based on the total relative levels of volatile organics in the environment.
Piezometer	A meter used to measure the pressure under earthen dams, foundations, boreholes and within aquifers.
Pitot Tube	Measuring device used to determine gas velocities in emission sources.
Plume	In air monitoring context, a feathery emission of gases, liquids and particulates from a point source such as a stack or vent.
PM2.5	Particles having an aerodynamic diameter ≤ 2.5 micrometers.
PM10	Particles having an aerodynamic diameter ≤ 10 micrometers.
Point Source	In air sampling, a fixed emission source such as a stack, vent, or discharge pipe.
Point Source (CWA Usage)	Any discernible, confined, and discrete conveyance, including but not limited to, any pipe, ditch, channel, tunnel, conduit, well, discrete fissure, container, rolling stock, concentrated animal feeding operation, landfill leachate collection system, vessel or other floating craft from which pollutants are or may be dis-

	charged. This term does not include return flows from irrigated agriculture or agricultural storm water runoff.
Poison Division/Class 2.3	Poisonous gases or liquids of such a nature that a very small amount of the gas, or vapor of the liquid, mixed with air is dangerous to life (Title 49 CFR 173.326).
Poison Division/Class 6.1	Demonstrated an oral LD50 of up to and including 50 mg/kg, or in inhalation LC50 of up to and including 2 mg/liter, or a general LD50 of up to and including 200 mg/kg; or is either classed as Poison Class B per Title 49 CFR 173.343, or qualifies as a Category I Pesticide per Title 40 CFR Part 162 excluding the corrosivity criteria.
Pollutant (CWA Usage)	Dredged spoil, solid waste, incinerator residue, filter backwash, sewage, garbage, sewage sludge, munitions, chemical wastes, biological materials, radioactive materials (except those regulated under the Atomic Energy Act), heat, wrecked or discarded equipment, rock, sand, cellar dirt, and industrial, municipal, and agricultural waste discharged into water. It does not mean: (a) sewage from vessels; or (b) water, gas, or other material which is injected into a well to facilitate production of oil or gas, or water. (See complete definition in 40 CFR 122.2).
Polychlorinated Biphenyl (PCB)	Any of 209 compounds or isomers of the biphenyl molecule that have been chlorinated to various degrees (includes monochlorinated compounds). PCBs are highly toxic to aquatic life, they persist in the environment for long periods of time, and they are biologically accumulative.
Precision	The degree of reproducibility of results, or the degree of mutual agreement among independent, similar, or repeated measurements.
Predictive Modeling of Air Pollution	Method of assessment based on choosing an emission model for a particular source type. Data are usually obtained from literature, estimation, or calculation. Generally used for screening.
Pressurized Sampling	A canister sampling procedure for collection of whole air samples. A metal bellows-type pump forces ambient air into an evacuated canister until the pressure reaches 15–30 psig.
Pretreatment Standards	
(CWA Usage)	Specific industrial operation or pollutant removal required prior to discharge of contaminated water to a municipal sewer.
Procedure	A set of systematic instructions for performing an operation.
Proficiency Testing	Determination of field or laboratory testing performance by means of inter-laboratory comparisons.
Protocol	Thorough, written description of the detailed steps and procedures involved in the collection of samples.

Public Water System (PWS)	A system for the provision to the public of piped water for human consumption, if such system has at least 15 service connections or regularly serves an average of at least 25 individuals daily at least 60 days of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control that are used primarily in connection with such system. A public water system is either a "community water system" or a "non-community water system" and may be "transient" or "non-transient."

Q

Qualitative	The identification, characterization, or description of materials.
Quality Assessment	The evaluation of data to determine if they meet the quality criteria required for a specific application.
Quality Assurance	An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that sampling and testing meet defined standards of quality with a stated level of confidence.
Quality Assurance Plan	A written plan that describes the methods and procedures that are required to assure the validity of sampling and analytical data.
Quality Control	The activity whose purpose is to measure and control the quality of sampling and testing so that it meets the needs of users and provides assurance that the appropriate level of confidence is achieved.
Quantitative	A measurable number, quantity or percent composition of a mixture. A number or amount of anything, either specified or indefinite.

R

Radioactivity	Atomic emission resulting from natural or artificial nuclear transformation. The energy of the process is emitted in the form of alpha, beta, or gamma rays by the decaying of the nuclei of the atom.
Radionuclide	A radioactive species of atom characterized by the constitution of its nucleus and hence by the number of protons, the number of neutrons, and the energy content.
Range	Spread of values calculated by subtracting the lowest value from the highest value. The extent of variation.
Raw Data	Any worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations, and or activities of a study and are necessary for the reconstruction and evaluation of the reports of that study. Raw data may include pho-

	tographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.
Reagent	Any substance used in a reaction for the purpose of detecting, measuring, examining, or analyzing other substances.
Reagent Blank (Method Blank)	A clean sample that contains any reagents used in the sample preparation and analysis procedure and processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the procedure.
Reagent Water	Water demonstrated to be free from the analyte(s) of interest and potentially interfering substances at the MDL for that analyte in the reference method or additional method. Reagent water may be defined as to the quality of the water to be used for analysis (see Distilled Water).
Real-Time Sampling	In air monitoring context, refers to field measurements that can be made with various instruments to quickly survey the area. Grab samples taken in the field over very short collection periods may also be referred to as real-time samples.
Reportable Quantity	The quantity specified in column 2 of the Hazardous Materials Table in Title 49 CFR 172.101, for any material identified by the letter “E” in Column 1 (Title 49 CFR 171.8), or any material identified by EPA on Table 117.3, Reportable Quantities of Hazardous Substance in Title 40 CFR 173. The letter “E” in column 1 (Title 49 CFR 172.101) identifies this material as a potential hazardous substances.
Representativeness	Correspondence between an environmental sample and the actual material that is present in the environment. The degree to which the data collected accurately reflect the universe of data from which they are drawn, or the degree to which samples represent true systems.
Reproducibility	The extent to which a method, test, or experience yields the same or similar results when performed on subsamples of the same sample by different analysts or laboratories. Usually expressed in terms of precision and accuracy.
Residue	As related to Title 49 CFR 171.8, residue is the hazardous material remaining in a packaging after its contents have been emptied and before the packaging is refilled, or cleaned and purged of vapor to remove any potential hazard. Residue of a hazardous material, as applied to the contents of a tank car (other than DOT Specification 106 or 110 tank cars), is a quantity of material no greater than 3 percent of the car’s marked volumetric capacity.

Retention Time	Period of time from the injection of a sample into a gas chromatography system until the point of maximum detector response for each substance.
Rodenticide	A chemical that is toxic to rodents such as mice and rats, and is approved by U.S. EPA for use for this purpose.

S

Salinity	A measure of the concentration of dissolved salts in a liquid.
Salvage Drum	A drum with a removable metal head that is used to store and transport damaged or leaking containers of hazardous materials (See Title 49 CFR 173.3.) (Also referred to as disposal or recovery drum.).
Sample Collection	The procedures involved in obtaining a representative portion of a material.
Samples	Portions of material such as soil, water, air, waste or contaminants extracted from the media that are representative of the material present in the environment.
Sampling	The techniques used for obtaining or extracting representative test samples from the environment.
Saranex®	A plastic fabric that is coated with polyethylene to resist penetration by water and chemicals. Used to make disposable coveralls that are used where there is the risk of splashes or exposure to liquids.
Secondary Maximum Contaminant Level (SMCL)	The maximum permissible level of a contaminant, defined in 40 CFR 143, in water that is delivered to the free flowing outlet of the ultimate user of a public water system.
Seep	A spot where a fluid (as water, oil, or gas) contained in the ground oozes slowly to the surface and often forms a pool.
Semivolatile Organic Chemicals (SVOCs)	Organic chemicals that have a vapor pressure range from 10-12-10-8 KPa. Chemicals that do not evaporate rapidly, but which will vaporize at temperatures below which they decompose.
Serial Dilution	Performing a series of dilutions on a substance to generate a calibration curve or to reduce the concentration of a substance to the measurable level.
Shelby Tubes	A type of sampling device used to obtain soil samples.
Sieve	A woven-like structure made of intersecting strands of wire or plastic, usually mounted in a steel frame. They are available in a wide range of sizes, weaves, and meshes from as coarse as 25 to as fine as 400.

Sludge	A water formed sedimentary deposit, usually a solid or semi-solid material that has settled out of a liquid.
Soil	The unconsolidated mineral and organic matter on the surface of the earth that has been subjected to and influenced by geologic and other environmental factors.
Soil Gas	Subsurface gas that may be generated by biological, chemical, and physical decomposition.
Soil Texture	A standardized description used to differentiate types of soil classes by geologists. Used to differentiate sand, sandy loam, loam, etc., relevant classifications can be found in the United Soil Classification Systems (USCS), Burmeister, and United States Department of Agriculture (USDA).
Soluble	Capable of being dissolved.
Solution	A spontaneously forming homogeneous mixture of two or more substances, retaining its constitution in subdivision to molecular volumes and having various possible proportions on the constituents.
Solvent	The component of a solution that is present in excess or that undergoes no change of state. A liquid capable of dissolving another substance.
Soot	Aerosol carbon particulates found in smoke from combustion processes of natural or manmade origin (0.01–1.0 µm diameter).
Sorbent	A solid material used to extract a chemical contaminant from a gas or a liquid.
Split Spoon Sampler	Soil sampling device used during drilling of boreholes to obtain soil samples at specific depths.
Standard Operating Procedure (SOP)	A written document that details an operation, analysis, or action whose mechanisms are thoroughly prescribed and that is commonly accepted for performing certain routine or repetitive tasks.
Steam Jenny	A piece of transportable equipment that generates low pressure steam; used for cleaning equipment such as drill rigs.
Storage	When used in connection with hazardous waste, means the containment of hazardous waste, either on a temporary basis or for a period of years, in such a manner as not to constitute disposal of such hazardous waste.
Storage Facility	Any facility used for the retention of hazardous waste prior to shipment or usage, except generator facilities (under Title 40 CFR) which is used to store wastes for less than 90 days, for subsequent transport.
Sub-Atmospheric Sampling	A canister sampling procedure for collection of whole air samples in which the canister is under vacuum. A grab sample can

	be collected by opening the inlet for 10 – 30 seconds, or a time-integrated sample can be collected over a period of up to 24 hours by using a flow-restrictive orifice.
Subsurface Soil	The soil more than a defined distance below grade and extending downward to the top of the seasonally high water table.
Summa Canisters	Metal canisters with a chrome-nickel oxide inside surface (Summa polishing process) used to collect whole air samples.
Support Zone	Uncontaminated area defined at a remediation site where administrative functions needed to keep site operations running smoothly are conducted.
Surface Soil	The soil extending from the surface to just above a defined distance below grade.
Surface Impoundment	Any natural depression or excavation and/or dike that is built into or upon the land, which is fixed, uncovered, and lined with soil or a synthetic material, and is used for treating, storing, or disposing wastes. Examples include holding ponds and aeration ponds.
Surveillance	Observation or supervision to gather information.
Swale	A natural or manmade low-lying or depressed and often wet stretch of land.
System Blank	A quality control sample or blank used to measure the background or baseline response in the absence of a sample.
Systematic Error	Error that usually results in a consistent deviation or bias in a final result.

T

Tedlar® Bag	Brand name for a collapsible container equipped with a resealable valve and used to collect and store gas samples.
Teflon®	Brand name for a type of plastic that is resistant to organic and most inorganic chemicals.
Tenax®	Brand name for a 2,6-diphenylene oxide polymer used as sampling media for volatile and semi-volatile organic chemicals.
Thermal Desorption	Use of heat to remove volatile and semi-volatile organic compounds from a solid sorbent.
Thermal Gradients	In air monitoring context, the result of uneven heating and cooling of the earth and surrounding air layers and the decrease of atmospheric pressure with vertical height.
Threshold	The level where the first effects occur; also the point at which a person just begins to notice an effect. (OSHA Usage).
Time Resolution of Sensors	Time required for a real-time sensor, recording in situ measurements, to reach 90 percent of the final response to an incremental change in contaminant concentration.

Topographical Factors	Natural surface features such as mountains, hills, valleys, lakes, and seas, and man-made features such as high-rise buildings, highways, and parking lots which influence the dispersion of air pollutants.
Topography	The geological or physical features of a particular place.
Total Matter	The sum of the particulate and dissolved matter.
Toxicity	The harmful effects of a chemical on some biological mechanism and the condition under which these effects occurs.
Toxicity Characteristic Leaching Procedure (TCLP)	Test procedure defined by EPA to determine whether a waste material placed in a landfill may contaminate groundwater.
Trace Analyte	The trace metal(s) or trace organic(s) which are to be determined in the sample of interest.
Trace Metals	Naturally occurring chemical elements that are everywhere in the environment and generally in very low concentrations (< 10 ppm). Examples include the 13 elements classified as priority pollutants by the EPA.
Trace Metal Grade	Reagents that have demonstrated to be free from the metal(s) of interest at the MDL of the analytical method to be used.
Trace Organics	Manmade (synthetic) or naturally occurring compounds composed of carbon, hydrogen, and oxygen that occur in low concentrations in the environment (<100 ppm). Examples are polyaromatic hydrocarbons from fossil fuel combustion, synthetic pesticides such as diazanon, and naturally occurring and manmade compounds such as dioxins and furans.
Traceability	The property of a sample or measurement whereby it can be related to appropriate international or national standards through an unbroken chain.
Transient Non-community Water Systems (TNCWS)	A non-community water system that does not regularly serve at least 25 of the same persons over 6 months per year.
Trip Blank	A quality control sample consisting of a clean sample matrix that is carried to the sampling site and transported to the laboratory for analysis without having been exposed to the sampling procedures. The purpose of the trip blank is to determine whether samples might have been contaminated during transport and storage.
Turbidity	Reduction in a liquid's transparency from the presence of particulate matter.
Turnaround Time	The amount of time in hours or days from sample receipt at the laboratory to delivery of final report.

Tyvek®	Brand name for a plastic fabric used to make disposable coveralls that help protect personnel from exposure.

U

Upper Explosive Limit (UEL)	Maximum concentration of a combustible gas or vapor in air that will explode when ignited.
Upwind Sample	A sample taken in air monitoring upwind of the source to ensure that the actual sample is not being contaminated or biased by other sources of emissions in the sampling area.

V

Vadose Zone	Groundwater located in the zone of aeration above the water table.
Vapor Pressure	The pressure characteristic at any given temperature of a vapor in equilibrium with its liquid or solid form. Higher values indicate higher volatility or evaporation rate.
Vapor Probe	That portion of an instrument that is used to collect a gas; the part of the instrument inserted into soil when conducting soil gas analysis.
Variance	Measure of the variability of a property or measurement. A statistical term describing the variation of values in a data set around some common value. Referred to as sample variance or population variance depending on the calculation procedure.
Verification	Confirmation by examination and provision of evidence that specified requirements have been met.
Vermiculite	Lightweight mineral made from expanded mica. May be used to pack sample containers for shipping.
Virus	An infectious agent composed almost entirely of protein and nucleic acids. Viruses can reproduce only within living cells.
Viscosity	Resistance to flow of a liquid or gas.
Visibility Measurement	Measurement of the greatest distance at which a dark object of reasonable size can be seen against the horizon sky. Measurement depends on degree of light transmission through the atmosphere and contrast of the object to the background. (Meteorological definition).
Volatile Organic Chemicals (VOCs)	Organic compounds that have a vapor pressure range >10–2kPa.
Volatilization	Process whereby solids and liquids pass into the vapor state at a given temperature.

W

Waiver	A document that relinquishes the holder from a requirement.
Waste Samples	Portions of waste that are representative of the material.
Water Solubility	The extent to which a compound dissolves in water.

Water Table	The seasonally high level of the surface of an aquifer.
Well Purging	Process in which the standing water is removed from a well and the well is allowed to refill with groundwater before a sample is collected.
Wet Collectors	In air sampling context, a collection device that uses a finely dispersed liquid to increase the size of aerosol particles.
Wetland	Those areas that are inundated or saturated by surface or groundwater at a frequency or duration sufficient to support, and that under normal circumstances do support, a prevalence of vegetation typically adapted for life in saturated soil conditions. Wetlands generally include lakes, swamps, marshes, bogs and similar areas such as sloughs, prairie potholes, wet meadows, prairie river overflows, mudflats, and natural ponds.
Whole Air Sample	An air sample taken without attempting to concentrate any specific pollutants.