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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING AND FIELD TESTING PROCEDURES MANUAL

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 1

INTRODUCTION

1.1 PURPOSE OF THIS MANUAL. This manual documents procedures for environmental sampling and field testing activities. It provides assistance to Navy shore activity personnel engaged in environmental sampling and field testing. Many of the baseline steps have been derived from approved/accepted regulatory programs. The intent of the manual is to promote consistency in the manner in which environmental samples are collected for analysis.

1.2 SCOPE. This manual applies to all U.S. Navy and Marine Corps shore activities internal and external to the U.S., and its territories and possessions, engaged in environmental self-monitoring. This manual does not apply to nuclear propulsion plants or associated nuclear support facilities, or practices related thereto which are under the cognizance of the Director, Naval Nuclear Propulsion Program.

Manual 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 manual, and this manual should be used as a guidance document only.

This manual applies to sampling and field testing performed principally for compliance assessment. It does not apply to sampling performed for the Navy Installation Restoration (IR) Program. Specific guidance for IR sampling is contained in the "Navy Installation Restoration Program Manual."

1.3 BACKGROUND. OPNAVINST 5090.1B, *Environmental and Natural Resources Program Manual*, provides Navy policy, identifies key statutory and regulatory requirements, and assigns responsibilities for complying with environmental laws/regulations, protecting the environment, conserving natural resources, preserving cultural and historic resources, and preventing pollution. MCO P5090.2, *Environmental Compliance and Protection Manual*, provides similar information to the Marine Corps. Requirements are complex, and there are serious legal as well as administrative concerns associated with failure to comply. Therefore, a disciplined approach is necessary to ensure success.

Environmental sampling and field testing actions are the focus of monitoring operations for compliance with regulations. The opportunity for error is great because of the variable conditions within the environment and the great variety of sampling equipment available for use. There is little tolerance for error since the validity of associated laboratory test results depends on sample integrity, and the results are the basis for many environmental decisions.

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/activities in this manner. At all times, success requires that workers 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/laboratory personnel interface
- Sampling equipment usage and maintenance requirements
- Sampling and field testing procedures
- Record/log keeping requirements
- Chain-of-custody requirements
- Personnel and equipment safety precautions
- Labeling, preservation, transportation, packaging, and shipping requirements
- Training and qualification requirements

This manual responds to the above concerns by providing clear, concise, and consistent guidance to personnel. The manual contents provide the basic framework to identify generic requirements pertaining to sampling, to the extent possible, given the diverse missions and sites of the U.S. Navy. The manual should augment and improve internal management of Navy shore facility environmental sampling programs. It is not intended to create any right or benefit, substantive or procedural, enforceable at law by any party against the Department of the Navy (DON), its officers, employees, or any person.

1.4 REQUIREMENTS FOR LABORATORY TESTING. The Navy is committed to operating ships and shore facilities in a manner compatible with the environment. An important element of the Navy's mission is, therefore, to prevent pollution, protect the environment, and comply with regulations established by Federal, state and local governments. To document the Navy's efforts in protecting the environment and to substantiate the Navy's compliance with environmental regulations, the Navy incurs substantial annual costs for environmental laboratory testing services. These services consist of two significant components: (1) assessing regulatory *compliance* of materials, systems, and processes, and (2) environmental *restoration* efforts.

1.4.1 Compliance Testing. The Navy has a continual need for laboratory testing services to evaluate compliance with regulatory limits defined for environmental pollutants. Regulatory compliance is critical to facility operations and fleet readiness. Principally, the Navy 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 Navy operates its facilities pursuant to regulatory requirements which address process discharge. Permits may be required for base support systems (drydocks, sewer, water, industrial waste treatment) and production processes (painting, degreasing, abrasive blasting, flushing). The Navy 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

The Navy's compliance with the procedural and substantive requirements of CERCLA and SARA, as well as regulations promulgated under these acts or by state law, are defined in the Installation Restoration (IR) Program.

Restoration testing is performed almost exclusively by commercial laboratories. Restoration work is accomplished using remediation contracts which sub-contract to commercial laboratories. The laboratories are approved by the Navy based on successfully completing a performance evaluation sample, providing an adequate Quality Assurance (QA) plan, undergoing an on-site audit, and submitting monthly progress reports thereafter. The process is presently undergoing modification to eliminate submission of monthly reports. These reports will be replaced by unannounced data package audits to ensure laboratories are submitting quality data.

RCRA also imposes some restoration testing requirements. This testing is performed by in-house and contract laboratories to support RCRA regulations.

1.5 MANUAL OVERVIEW. Chapter 1 summarizes the manual and gives direction for its use.

Chapter 2 provides **Sampling Personnel** a summary of the laws, regulations, and policies that require samples to be taken and then analyzed by the laboratory. **Appendix A** - *Bibliography of EPA Publications* is referenced in this chapter to provide relevant references for further information.

Chapter 3 provides an overview of the sampling program in general. It 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 EPA for each sampling event. A reference to **Appendix B** - *Training Sources* is referenced in this chapter to provide a guide to some relevant field sampling training.

Appendix C - *Health and Safety Plan Review* is referenced to provide a thorough review checklist for situations when a site specific “Health and Safety Plan,” as required by 29 CFR 1910.120, is necessary.

Appendix D - *NAVOSH Reference List* is referenced to provide pertinent hazard specific information resources which compliance sampling personnel and cognizant health and safety professionals should be aware of during the risk management process.

To **Sampling Personnel**, Chapters 4 through 15 represent the "core" of the manual by providing the procedures and requirements of field sampling. A single sampling run will involve the following sections (See **Figure 1-1)**:

- **Chapter 4** - *Common Sampling Procedures*
- **Chapter 5 (or 6, 7,..., 14)** - *Soil Sampling (or Sediment Sampling, Surface Water Sampling, ...Field Testing)*
- **Appendix H** - *Requirements for Sample Containers, Preservation, and Holding Times*
- **Appendix I** - *Requirements for Collection of Quality Control Samples*
- **Appendix F** - *DOT Shipping Requirements*
- **Chapter 15** - *Guidelines for Requesting Laboratory Testing*

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

- **Table of Contents** that lists and provides page numbers for all major sections and subsections
- **List of Figures** and **List of Tables** that lists and provides page numbers for all figures and tables
- **Tabs** to quickly locate all major sections
- **Glossary of Terms** and **Acronyms** to define commonly used words and abbreviations
- **Index** to locate key words and subjects throughout the manual
- **Table of References** to provide sources of information used throughout the manual and some additional sources of valuable information

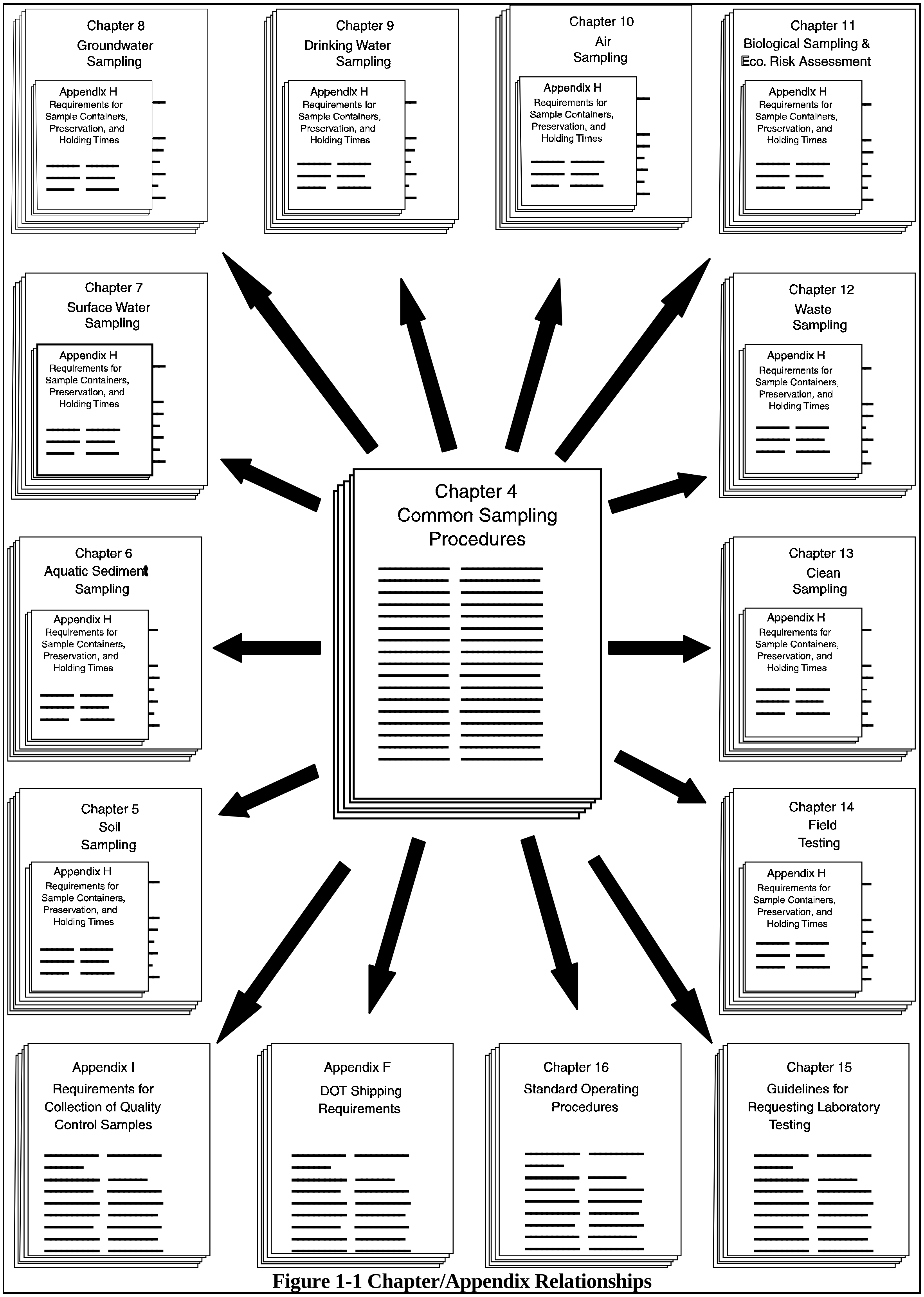

Figure 1-1 Chapter/Appendix Relationships

NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 2

ASSOCIATED STATUTORY AND REGULATORY REQUIREMENTS

2.1 PURPOSE. This chapter provides a synopsis of the major legislation, regulations, and policies that require environmental compliance samples to be collected and analyzed.

2.2 LAWS. The U. S. Congress and state legislatures have enacted numerous laws that authorize government agencies to take actions to protect the environment. These laws establish general government policies and goals. They generally leave the details of the required actions, including environmental sampling and analysis requirements, to the judgment of the agencies which establish the regulations and the guidance.

The following is a partial list of laws that establish requirements for environmental sampling and testing. Each of these laws leads to specific requirements and protocols that should be followed by sampling personnel to ensure a compliant environmental assessment program. Applicable major laws are summarized by media in **Table 2-1**.

2.2.1 Federal Environmental Laws. The following is a summary of major Federal laws.

2.2.1.1 National Environmental Policy Act (NEPA). In enacting NEPA, Congress established the basic national charter for protection of the environment. NEPA serves as an ``umbrella", embracing all Federal decisions, even when the Federal agency involved is not one with distinct environmental responsibilities. NEPA states environmental policy, sets goals, and provides the means for carrying out policy. Two basic tenets of NEPA are:

- Procedures must be in place to ensure that environmental information is available to decision makers and citizens before decisions are made and major Federal actions are taken.
- Planning should identify and assess reasonable alternatives to proposed actions to avoid or minimize environmental adverse effects.

2.2.1.2 Resource Conservation and Recovery Act (RCRA). The Resource Conservation and Recovery Act (RCRA), which amended the Solid Waste Disposal Act, regulates the management of solid waste and hazardous waste to protect public health and the environment. RCRA outlines the specific requirements for managing hazardous substances and defines permitting requirements for treatment, storage, and disposal facilities. RCRA also provides that substances identified as hazardous wastes be tracked with a "cradle-to-grave" manifest system. RCRA makes generators of hazardous substances responsible for them forever.

In 1984, Congress enacted the **Hazardous and Solid Waste Amendments (HSWA)** to RCRA. Major impacts of HSWA include:

- Underground storage tank requirements, including technical (corrosion protection, spill/overflow protection, leak detection), closure, reporting, and record keeping
- Land disposal requirements, which impose strict performance requirements on land disposal facilities
- Land ban requirements, which scheduled a phased-in "land disposal ban" for hazardous wastes

The HSWA required EPA to ban the land disposal of hazardous wastes unless they were first treated, so that failure of the disposal facility would not result in environmental damage. Land disposal includes landfills, surface impoundments (ponds), waste piles, injection wells, underground mines or caves, and concrete vaults or bunkers.

Additional amendments in 1988 established a requirement for tracking medical wastes.

2.2.1.3 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). CERCLA, also known as **Superfund**, was enacted to deal with environmental hazards caused by abandoned chemical dumps and past hazardous waste management practices. Under CERCLA, all property owners and waste generators who sent wastes to a site can be held liable for cleanup costs. Where no private responsible parties can be found, EPA will pay for the cleanup using money from **Superfund**, a fund

Table 2-1 General Applicability of Federal Environmental Laws

Environmental Media	Federal Environmental Legislation				
	CAA	CWA	SDWA	RCRA	TSCA
Waste Water, Sewage Sludge		X		X	
Ground Water			X	X	
Drinking Water		X	X		
Air	X			X	X
Stack Gas	X			X	
Soil, Sludge				X	X
Solid Waste				X	X
Waste Oil				X	

accumulated from taxes assessed on the manufacture of certain chemicals.

CERCLA requirements are addressed separately as part of the Navy Installation Restoration (IR) Program. CERCLA was amended by the **Superfund Amendments and Reauthorization Act (SARA) of 1986**. SARA required EPA to promulgate revisions to the **National Oil and Hazardous Substances Pollution Contingency Plan (NCP)**.

Title III of SARA is also known as the **Emergency Planning and Community Right-to-Know Act (EPCRA)** and has four major parts:

- Emergency planning
- Emergency notification
- Community right-to-know
- Toxic chemical release reporting

SARA also requires that all facilities using a listed toxic chemical in amounts exceeding threshold quantities report the quantity of emissions and off-site disposal of the chemical to EPA on an annual basis.

2.2.1.4 Federal Water Pollution Control Act (FWPCA). FWPCA of 1972 authorized EPA to protect surface water (streams, ponds, lakes, harbors, etc.) to achieve a national goal of fishable, drinkable, and swimmable surface water. Among the provisions of FWPCA are the requirements for:

- Non-point source pollution abatement plans to be consistent with EPA regulations
- A permit for any discharge to a navigable waterway **(National Pollution Discharge Elimination System NPDES)**. This permit system establishes limits on the amount of contaminants that can be present in discharged water
- A permit from the Army Corps of Engineers for any discharge of dredge or fill material into navigable waters and wetlands

The 1977 **Clean Water Act (CWA)** amended FWPCA to ensure that toxic chemicals are controlled through effluent guidelines, permits, and water quality standards, and that discharges are treated prior to their release to surface waters. The required treatment standards included:

- **Best Conventional Technology (BCT)**, which essentially required secondary treatment by sewage treatment plants that handled conventional pollutants generally found in domestic discharges
- **Best Available Technology Economically Achievable (BATEA)**, for treatment of waste waters containing priority toxic pollutants
- **Best Available Technology (BAT)**, for the treatment of water contaminated with chemicals other than listed priority toxic or conventional pollutants

2.2.1.5 Toxic Substance Control Act (TSCA). TSCA gave EPA the authority to regulate the manufacture and use of chemicals to the extent that such authority had not previously been established. Under TSCA, EPA required manufacturers and importers to report all chemicals manufactured in or imported to the U. S., and can require manufacturers to report any new uses of existing chemicals and submit any available data on the toxicity of chemicals. EPA can require that additional toxicity testing be performed to demonstrate that specific chemicals are not hazardous.

TSCA also required that EPA ban most uses of **polychlorinated biphenyls (PCBs)**, and regulate the marking, storage, transportation, and disposal of PCBs. EPA also regulates **asbestos, dioxins and furans** under TSCA provisions.

2.2.1.6 Safe Drinking Water Act (SDWA). SDWA protects drinking water supplies by establishing contaminant limitations and enforcement procedures. EPA has published two types of standards as follows:

- *Primary* drinking water standards to protect public health
- *Secondary* drinking water standards to protect public welfare and address aesthetic concerns

SDWA requires each state to adopt a program to protect wells within its jurisdiction from contamination. States have the primary responsibility of enforcing compliance of national primary drinking water standards, sampling, monitoring, and notification requirements.

2.2.1.7 Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA). FIFRA authorized EPA to prevent environmental pollution from pesticides, insecticides, fungicides, and rodenticides through product registration and applicator certification. The registration of all pesticide products by EPA results in label instructions on each container for use, storage, and disposal. Label instructions are legally applicable to all users. Under FIFRA, EPA is required to accept certain pesticides under recall for safe disposal. It is unlawful to purchase, distribute, or use any pesticide not having an EPA registration number, or for which registration has been canceled or suspended, or to apply, store, or dispose of any pesticide or container in any manner inconsistent with applicable regulations. **FIFRA** was amended in 1972 by the **Federal Environmental Pesticide Control Act**.

2.2.1.8 Clean Air Act (CAA). The CAA authorized EPA to establish a program of air pollution research, regulation, and enforcement activities. Under the CAA, the primary responsibility for preventing and controlling air pollution at its source rested with state and local governments. There was, however, a strong mandate that EPA take action when states do not fulfill their responsibilities.

The CAA establishes environmental standards for air quality which are prescribed as **National Ambient Air Quality Standards (NAAQS)**. The two types of standards are:

- *Primary* air quality standards to protect human health
- *Secondary* air quality standards to prevent damage to property, animals, vegetation, crops, and visibility

The health and other environmental effects of pollutants are delineated in criteria documents that are the basis for these standards. There are presently NAAQS for:

- Particulate matter (10 microns)
- Sulfur oxides
- Nitrogen oxides
- Carbon monoxide
- Ozone
- Lead

Other controls covered under the CAA included federally prescribed national emission standards for new motor vehicles and selected new stationary sources.

The CAA was substantially strengthened by the CAA Amendments of 1990 to include:

- Acid rain reduction, principally focused at utilities and stationary sources
- Increased regulation of volatile organic compounds (VOCs), carbon monoxide (CO) emissions, nitrous oxides (NOx), and other pollutants which contribute to ozone pollution
- Increased regulation on the emission of hazardous air pollutants (air toxins)
- Requirement to use clean burning fuels in mobile sources (trucks, cars, etc.)
- Increased enforcement with fines and penalties

2.2.1.9 Federal Facility Compliance Act (FFCA). The FFCA expands the enforcement authority of Federal and state regulators with respect to solid and hazardous waste management at Federal facilities. The FFCA requires Federal facilities to pay any nondiscriminatory fees or service charges assessed in connection with a Federal, state, interstate, or local solid or hazardous waste regulatory program. The FFCA also waives sovereign immunity for Federal facilities under solid and hazardous waste laws by allowing states to fine and penalize them for violations.

2.2.1.10 <u>Occupational Safety and Health Act (OSHA)</u>. The Department of Defense and the Navy have adopted the Occupational Safety and Health Administration (OSHA) standards as required under Section 6 of the Occupational Safety and Health Act, with provision for alternates to the OSHA standards, supplemental standards, other special standards, and exceptions for military unique equipment, systems, and operations as contained in DOD instruction 6055.1.

The relationship between OSHA standards and the NAVY Occupational Safety and Health Program is detailed in Chapter 16 of OPNAVINST 5100.23D, *Navy Occupational Safety and Health (NAVOSH) Program Manual*. OPNAV instructions, particularly the contents of OPNAVINST 5100.23D and any instructions issued by the command having specific technical cognizance or assigned responsibility in OPNAVINST 5100.8G and approved by the Chief of Naval Operations, are considered to be Navy Occupational Safety and Health (NAVOSH) standards. Some OSHA standards which may be specifically applicable to environmental compliance monitoring procedures are listed in **Section 2.3.3**.

2.2.2 <u>Federal Transportation Laws</u>. The definitions for shipping are set by the **Department of Transportation**, and are different from the definitions set by EPA. EPA regulates hazardous substances and hazardous wastes, but does not regulate hazardous materials.

2.2.3 <u>State Environmental Laws</u>. Discussion of specific state environmental laws is beyond the scope of this manual. However, most of the Federal environmental laws either assign the primary compliance responsibility to the states or provide mechanisms for transferring EPA authority to states. In general, states can impose more stringent requirements than those imposed by EPA, but are not allowed to set less stringent requirements. Therefore, knowledge of the state environmental requirements applicable to a particular facility is imperative when planning a sampling and analysis program.

2.3 <u>REGULATIONS</u>. Regulations are the enforceable requirements that agencies promulgate in order to implement laws. In general, sampling and analysis requirements are regulatory and are not set directly by associated laws. In terms of actually planning work, knowledge of the regulations is much more important than knowledge of the laws.

2.3.1 <u>U. S. EPA regulations</u>. EPA regulations are published as **Title 40 of the Code of Federal regulations (40 CFR)**. **40 CFR** actually consists of 15 volumes. Individual regulations are very detailed in specifying sampling methods, including both sampling equipment and procedures. The following is a guide to some of the sections of **40 CFR** that are relevant to sampling and analysis programs:

2.3.1.1 <u>Air Programs (Parts 50-99)</u>.

50 **National Ambient Air Quality Standards.**

58 **Ambient Air Quality Surveillance Regulations**. Subpart C specifies air quality monitoring methods required under the Clean Air Act

60 **Regulations on New Source Performance Standards (NSPS).** Specifies the maximum air discharge limits for various industries. The appendices include several hundred pages of detailed stack sampling methods.

61 **National Emission Standards for Hazardous Air Pollutants (NESHAPS).**

2.3.1.2 <u>Water Programs (Parts 100-149)</u>.

112 **Regulations on Oil Pollution Prevention.** Establishes procedures, methods, equipment and other requirements to prevent the discharge of oil from non-transportation related onshore and offshore facilities into or upon the navigable waters of the U. S. or adjoining shorelines. Section 112.7 lists guidelines for the implementation of a Spill Prevention Control and Countermeasure Plan, and includes directions on testing and inspection of containment equipment.

116 **Regulations on Hazardous Substances.** Part 116 lists hazardous substances under Section 311(b)(2)(A) of the FWPCA. Part 117 includes the table used to determine when a hazardous substance needs to be reported.

121 **Regulations on Implementing FWPCA/CWA.** Part 121 describes regulations regarding state certification of activities requiring Federal licenses or permits. These licenses and permits are granted by a Federal government agency to conduct any activity which may result in discharge into the navigable waters of the United States. Part 122 specifically details the NPDES permit program.

125 **Regulations on Criteria and Standards for the National Pollution Discharge Elimination System.** Establishes criteria and standards for the imposition of technology-based treatment requirements in permits under Section 301(b) of the CWA, including the application of EPA-promulgated effluent limitations and case-by-case determinations of effluent limitations under

Section 402(a)(1) of the CWA. 40 CFR 125.62 discusses the general requirements of the establishment of a monitoring program.

129 **Toxic Pollutant Effluent Standards.** Regulates the maximum allowable discharges of toxic pollutants, including pesticides and PCBs, into navigable waterways.

136 **Guidelines for Establishing Test Procedures for the Analysis of Pollutants.** Establishes proper sampling and analysis protocols for wastewater pursuant to compliance with the NPDES Program.

141 **National Drinking Water Regulations (Parts 141-143).** Establishes primary and secondary drinking water regulations pursuant to Section 1412 of the Public Health Service Act, as amended by the Safe Drinking Water Act (Pub. L. 93-523), and related regulations applicable to public water systems. Subpart C describes routine monitoring and analytical requirements for various types of sampling. Examples include: coliform sampling, turbidity sampling, inorganic chemical sampling, organic chemicals (other than total trihalomethanes) sampling, and radioactivity determination.

144 **Underground Injection Control Program.** This part sets forth requirements for the Underground Injection Control (UIC) program promulgated under Part C of the SDWA and, to the extent that they deal with hazardous waste, RCRA. To the extent set forth in Part 145, each State must meet these requirements in order to obtain primary enforcement authority for the UIC program in that State.

2.3.1.3 Pesticide Programs (Parts 150-189).

150 **Regulations for Pesticide Programs (Parts 150-186).** The purpose of Part 158 is to specify the types and minimum amounts of data and information that EPA requires in order to make regulatory judgments about the risks and benefits of various kinds of pesticide products under the criteria set forth in FIFRA.

2.3.1.4 Noise Abatement Programs (Part 201).

2.3.1.5 Ocean Dumping, Dredge and Fill (220-233).

220 **Ocean Dumping Regulations and Criteria (Parts 220-229).** The disposal site monitoring program under the FWPCA is described in Section 228.9. The program is designed to evaluate the impact of disposal on the marine environment by referencing monitoring results to a set of baseline conditions.

230 **Interim Regulations on Discharge of Dredged or Fill Material into Navigable Waters (Parts 230-233).** The purpose of these guidelines is to restore and maintain the chemical, physical, and biological integrity of waters of the United States through the control of discharges of dredged or fill material. Subpart G of this part describes the evaluation and testing in support of this process. It provides guidance in determining which test and/or evaluation procedures are appropriate in a given case.

2.3.1.6 Solid Waste (240-299).

240 **Guidelines for the Thermal Processing of Solid Wastes and for the Land Disposal of Solid Wastes (Parts 240-241)**. These parts list the requirements and recommended procedures for thermal processing and land disposal of solid waste. Requirements for monitoring and data recording are also specified in these parts.

259 **Standards for the Tracking and Management of Medical Waste.**

260 **Regulations Implementing RCRA (Parts 260-270).** These regulations include identification and listing of hazardous wastes and standards applicable to generators of hazardous waste. Monitoring plans are also described in order to support petitions to allow land disposal of prohibited wastes listed under Subpart C of Part 268. Part 270 details the EPA-administered permit program for hazardous waste.

280 **Underground Storage Tanks (USTs).** The requirements of this part apply to all owners and operators of an Underground Storage Tank System. Section 280.34 describes details for document submission, testing, and monitoring by the owner or operator pursuant to Section 9005 of Subtitle I of RCRA. Methods described include: release detection for tanks and piping, release detection record keeping, and reporting and cleanup of spills and overfills.

2.3.1.7 Superfund, Emergency Planning, and Community Right-to-Know Programs (300-399).

302 **Designation, Reportable Quantities, and Notification Requirements for Hazardous Substances Under CERCLA.** This regulation sets forth reportable quantities for hazardous substances designated under Section 311(b)(2)(A) of the CWA. Section 302.8 requires notification of any release of a hazardous substance listed under CERCLA.

311 **Worker Protection.**

355 **Emergency Planning and Notification.**

372 **Toxic Chemical Release Reporting Requirements.** This part lists requirements for the submission of information relating to the release of toxic chemicals under Section 313 of Title III of SARA. The information collected under this part is intended to inform the general public about releases of toxic chemicals, to assist research, to aid in the development of regulations, guidelines, and standards, and for other purposes. Section 372.85 includes toxic chemical release reporting forms and instructions.

2.3.1.8 Effluent Guidelines and Standards (400-471).

401 **General Provisions.**

403 **Effluent Guidelines and Pretreatment Regulations for Waste Water (Parts 403-471).** These parts list effluent limitations guidelines for existing sources, standards of performance for new sources and pretreatment standards for new and existing sources under the FWPCA. Point sources of discharges of pollutants are required to comply with these regulations and with permits issued by states or the EPA under the National Pollutant Discharge Elimination System. Toxic pollutants designated under FWPCA are listed in these parts as well as specific effluent limitations. For example, Section 401.17 describes pH effluent limitations under continuous monitoring and Section 413 describes monitoring requirements for electroplating.

2.3.1.9 Sewage Sludge (501-503).

503 **Standards for the use or disposal of sewage sludge.** This part establishes standards, which consist of general requirements, pollutant limits, management practices, and operational standards, for the final use or disposal of sewage sludge generated during the treatment of domestic sewage in a treatment works. Standards are included for sewage sludge applied to the land, placed on a surface disposal site, or fired in a sewage sludge incinerator. Also included are pathogen and alternative vector attraction reduction requirements for sewage sludge applied to the land or placed on a surface disposal site. In addition, the standards include the frequency of monitoring and record keeping requirements.

2.3.1.10 Toxic Substances Control Act (TSCA) (700-789).

761 **PCB Manufacturing, Processing, Distribution in Commerce, and Use Prohibitions.** This part establishes requirements for, and prohibitions of, the manufacture, processing, distribution in commerce, use, disposal, storage, and marking of PCBs and PCB items.

763 **Asbestos.**

2.3.2 U. S. Department of Transportation (DOT) Regulations. The DOT regulations are published in Title 49 of the Code of Federal Regulations (49 CFR). The Hazardous Materials regulations are in 49 CFR Parts 100 through 199. Parts 171 through 179 include instructions as to when hazardous waste manifests are required and the proper shipping names and shipping labels that must be used for hazardous materials. 49 CFR 172 provides some exemptions for regulating transportation of preserved samples (e.g., reference Table II of 40 CFR 136).

2.3.3 OSHA Regulations.

29 CFR 1910.28	Safety requirements for scaffolding
29 CFR 1910.95	Occupational noise exposure
29 CFR 1910.96	Ionizing radiation
29 CFR 1910.97	Nonionizing radiation
29 CFR 1910.120	Hazardous Waste Operations and Emergency Response
29 CFR 1910.133	Eye and face protection
29 CFR 1910.134	Respiratory protection
29 CFR 1910.146	Permit-required confined spaces
29 CFR 1910.147	Control of hazardous energy (lockout/tagout)
29 CFR 1910.156	Fire brigades
29 CFR 1910.157	Portable fire extinguishers
29 CFR 1910.252	General requirements
29 CFR 1910.1030	Bloodborne pathogens
29 CFR 1910.1200	Hazard communication
29 CFR 1910.1450	Occupational exposure to hazardous chemicals in laboratories
29 CFR 1926 Subpart L	Scaffolding

2.3.4 State Environmental Regulations. Discussion of specific state environmental regulations is beyond the scope of the manual. However, each state promulgates regulations to implement its environmental laws. In many cases, state regulations are a repeat of the EPA regulations, but this should not be assumed since the states have the authority to impose more stringent requirements than those imposed by EPA.

2.4 GUIDANCE AND PROCEDURES. EPA, state environmental agencies, other government agencies, and private publishers have prepared books describing in useful detail how to prepare Field Sampling Plans (FSPs), how to collect environmental samples, and how to analyze the samples.

2.4.1 U. S. EPA. EPA has prepared numerous procedure and guidance manuals that are relevant to environmental sampling programs. Procedures are also contained in the aforementioned regulations. **Appendix A** contains a short bibliography of useful EPA publications.

2.4.2 Occupational Safety and Health References. A listing of relevant Occupational Safety and Health references is provided in the **Table of References** at the end of the manual.

2.4.3 State. A number of states have prepared guidance documents that cover environmental sampling and analysis. State guidance should be considered in collecting and analyzing samples to comply with state requirements.

2.4.4 Other Standards. The following manuals are available from the Corps of Engineers:

- *Soil Sampling.* CEO EM 1110-2-1907
- *Quality Control of Water Quality Field Sampling.* CEO TEL 1110-2-252

The **American Society for Testing and Materials (ASTM)** has prepared a number of standard procedures that can be used to support the preparation of Sampling and Analysis Plans (SAP). The following is a list of some of the standards prepared by ASTM:

D420: *Standard Guide to Site Characterization for Engineering, Design, and Construction Purposes*
D888: *Standard Test Methods for Dissolved Oxygen in Water*
D1293: *Standard Test Method for pH in Water*
D1452: *Standard Practice Method for Soil Investigation and Sampling by Auger Borings*
D1586: *Standard Method for Penetration Test and Split Barrel Sampling of Soils*
D1587: *Standard Practice for Thin-Walled Tube Geotechnical Sampling of Soils*
D3370: *Standard Practice for Sampling Water*
D3694: *Standard Practice for Preparation of Sample Containers and for Preservation of Organic Constituents*
D3975: *Standard Practice for Development and Use of Samples for Collaborative Testing of Methods for Analysis*
D4448: *Standard Guide for Sampling Ground Water Monitoring Wells*
D4536: *Standard Test Method for High-Volume Sampling for Solid Particulate Matter and Determination of Particulate Emissions*
E1370: *Standard Guide for Air Sampling Strategies for Worker and Workplace Protection*
F1084: *Standard Guide for Sampling Oil/Water Mixtures for Oil Spill Recovery Equipment*

2.4.5 General Reference Books. A number of books on environmental sampling and analysis have been published commercially.

NOTE:

> Citation of specific educational or reference material does not constitute approval or endorsement of the publication. Rather, it is intended to provide an example of the type of publication.

2.5 NAVY/DEPARTMENT OF DEFENSE (DOD) INSTRUCTIONS AND TECHNICAL PUBLICATIONS.

2.5.1 OPNAVINST 5090.1B, *Environmental and Natural Resources Program Manual*. OPNAVINST 5090.1B provides Navy policy, identifies key statutory and regulatory requirements, and assigns responsibility for the management of Navy programs for:

- Compliance with current laws and regulations for the protection of the environment and the preservation of natural, cultural, and historic resources
- Cleanup of waste disposal sites
- Conservation of natural resources
- Pollution prevention
- Technology

OPNAVINST 5090.1B is divided into 25 chapters. Chapter 1 provides the scope, policy, organization, funding, and responsibilities applicable to the environmental and resources protection program. Subsequent chapters contain requirements and responsibilities for specific program areas (e.g., Clean

Air Ashore, Clean Water Ashore, Pesticide Compliance Ashore, Storage Tanks, and Noise Prevention Ashore). Each chapter discusses associated statutory and administrative requirements, including laws, 40 CFR, and DOD/OPNAV directives. Command responsibilities are also discussed.

Chapter 25 (to be issued in the near future) contains policy and guidance applicable to sampling and laboratory testing for environmental regulatory determinations for Navy shore facilities. It identifies requirements and responsibilities to ensure that measurements and collected data are accurate, that they meet requisite quality objectives, and are appropriate for use by the Navy in making decisions concerning the environment.

2.5.2 OPNAVINST 5100.23D, *Naval Occupational Safety and Health (NAVOSH) Program Manual*.

OPNAVINST 5100.23D is the primary reference document describing the NAVOSH Program. All elements of this reference apply to protecting the health and safety of NAVY DOD environmental compliance personnel. Elements which merit special attention relevant to environmental sampling include, but are not limited to:

- *Chapter 6: Training.* Establishes OSH training policy for Navy personnel.
  - Appendix 6-A, *Occupational Safety and Health Training Requirements*, requires that all personnel receive special training before working with toxic chemicals or hazardous waste.
  - Appendix 6-B, *Hazard Communication Training,* covers training specific to toxic chemicals and establishes the Navy Hazard Information System (HIS) as the source of information on chemicals.
- *Chapter 8: Occupational Health.* Establishes medical surveillance protocols.
- *Chapter 15: Respiratory Protection.* Establishes requirements and responsibilities for an ashore respiratory protection program. Requires that a certified Respiratory Protection Program Manager (RPPM) shall be appointed in writing by the commanding officer or officer in charge.
- *Chapter 20: Personal Protective Equipment (PPE).* Establishes basic requirements for personal protective equipment.
- *Chapter 27: Confined Space Entry Program.* Requires each major facility to establish a Confined Space Program and appoint a Confined Space Program Manager. A special Confined Space Entry Permit is required prior to entry into any designated confined space.

2.5.3 Navy Environmental Health Center Technical Publications. The Navy Environmental Health Center (NEHC) has prepared a number of technical publications that can be used to support the preparation of Sampling and Analysis Plans (SAP). The following is a list of some of the publications prepared by NEHC:

NEHC-TM90-1:	*Occupational Medicine Field Operation Manual*, March 1990
NEHC-TM90-2:	*Polychlorinated Biphenyls (PCBs), Polychlorinated Dibenzofurans (PCDFs), and Polychlorinated Dioxins (PCDDs)*, May 1990
NEHC-TM90-3:	*Emergency Medical Treatment Protocols for Hazardous Materials*, August 1990
NEHC-TM91-1:	*Manmade Vitreous Fibers*, October 1990
NEHC-TM91-2:	*Industrial Hygiene Field Operations Manual*, Revised March 1993
NEHC-TM91-3:	*Industrial Hygiene Sampling Guide for Consolidated Industrial Hygiene Laboratories*, Revised May 1993
NEHC-TM91-5:	*Medical Surveillance Procedures Manual and Medical Matrix (Edition 4)*, September 1991
NEHC-TM91-6:	*Advanced Composite Materials*, September 1991
NEHC-TM92-2:	*Reproductive Hazards in the Workplace: A Guide for Occupational Health Professionals*, May 1992
NEHC-TM92-5:	*Ultraviolet Radiation Guide*, April 1992
NEHC-TM92-6:	*Prevention and Treatment of Heat and Cold Stress Injuries*, June 1992
NEHC-TB-1:	*Acceptable Respirators for Use with Ambient Air Breathing Apparatus (AABA)*, October 1988

2.5.4 MCO P5090.2, Environmental Compliance and Protection Manual. MCO P5090.2 provides Marine Corps policy, identifies statuary and regulatory requirements, and assigns responsibilities for management of Marine Corps programs for the following:

- protection of human health and the environment
- compliance with appropriate laws
- remediation of past contamination
- pollution prevention
- preservation of natural, cultural, and historic resources

MCO P5090.2 presents overall policy and program management in the opening five chapters, followed by environmental media specific and technical issue chapters. Each chapter is broadly divided into three parts: Background, Marine Corps Policy, and Responsibilities. Appendix A includes discussions of the laws and other sources which generate the requirements, regulations, standards, mandates, policy, and guidance that form the basis of this manual.

2.6 PERMIT REQUIREMENTS. A significant amount of routine sampling is performed to demonstrate compliance with the requirements of operating permits at sewage treatment plants, stack emission permits, NPDES permits, etc. In each case, the permit writer and the regulated entity both negotiate what samples will be collected, how often samples will be collected, and for what the samples will be analyzed. The requirements of the permit supersede all other regulations and technical guidance.

The extent of quality control is dependent on regulatory program, matrix, and media. A Sampling and Analysis Plan (SAP) is not required by law for compliance monitoring, but is recommended for all compliance sampling and testing events to assure proper Quality Assurance (QA). The SAP and Quality Assurance Plan (QAP) address the details of sampling and analysis to ensure ongoing consistency and quality control (QC) of the sampling process. The QAP lists the QC data needed to demonstrate that project design meets the compliance requirements for both sampling QC and laboratory QC. In general, permits often contain specific details related to laboratory requirements such as sample quantities, preservation, holding times, test methods, and quality control. These details must be specified to assure that the sample meets laboratory requirements.

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 3

SAMPLING AND FIELD TESTING PROGRAM OVERVIEW

3.1 PURPOSE. This chapter discusses the following topics:

- Planning a sampling and analysis program for compliance monitoring
- Preparing a Sampling and Analysis Plan
- Organizing the sampling team to collect the samples and perform required field measurements and tests

3.2 NEED FOR PROPER PLANNING. The collection of useful environmental data is a collaborative effort involving the regulator, the program manager, field sampling personnel, the laboratory personnel, and the facility health and safety personnel.

3.2.1 Program Manager. The Program Manager (however named) defines the environmental data that is needed to meet project objectives, the samples that must be collected and analyzed to provide the data, and the quality assurance procedures that must be implemented to assure the required reliability of the data. The Program Manager is responsible for preparing the **Sampling and Analysis Plan (SAP)** by coordinating the input of the laboratory into the **Quality Assurance Plan (QAP)**, the field sampling personnel into the **Field Sampling Plan (FSP)**, and coordinating with cognizant safety and health professionals to ensure an appropriate site specific risk management process is completed in addition to maintaining a functional Navy Occupational Safety and Health (NAVOSH) program for NAVY DOD environmental compliance sampling and laboratory personnel.

The Program Manager (however named) is the person who has a need to know about the presence and concentration of toxic chemicals or pollutants to satisfy a regulatory program. This information may be required by the United States Environmental Protection Agency (EPA), state permit, local agency permit, to determine whether a material is classified as hazardous waste, or for the determination of safe drinking water. To obtain the necessary information, the Program Manager:

- Determines the applicable regulatory program(s) for compliance
- Assembles team members to meet the needs of the sampling and testing program
- Requests field sampling and laboratory analysis and specifies in writing exactly how the laboratory is to analyze each sample and what information and documentation is required from the laboratory
- Funds the required field and laboratory work, including required specialty personnel, materials and equipment
- Specifies in writing to field sampling personnel exactly:
    - 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
    - Any hazards which may be encountered by the sampling personnel, and procedures which must be followed to protect against these hazards

3.2.2 Sampling Personnel. Sampling personnel must:

- Furnish the Program Manager with suggested sampling methods to meet the project requirements
- Ensure that all involved field personnel have received the specified health and safety training and medical surveillance
- Ensure that all specified equipment is available and functional
- Perform the field measurements and tests and collect the samples as specified in the FSP
- Inform the laboratory as far in advance as possible as to when the samples will be shipped and the quantities and types of analyses required
- Submit a copy of all Field Log/Field Note entries to the Program Manager, or distribute as specified in the FSP
- Comply with all of the chain of custody and documentation requirements specified in the FSP and submit copies to the Program Manager, or distribute as specified in the FSP
- Complete the shipping manifest as required by the FSP, and furnish a copy to the Program Manager, or distribute as specified in the FSP
- Ensure explosive certifications are obtained, when required

- Support the activity NAVOSH Program and participate in the risk management process to ensure site specific safety and health hazards are identified and appropriate controls applied where needed (Supervisors must apply an appropriate level of supervision to ensure the risk management process is effective.)

3.2.3 Laboratory Personnel. The laboratory must:

- Review the analytical requirements to ensure that the laboratory can in fact perform the analyses and meet the required detection limits
- Furnish the Program Manager with information on laboratory detection limits for each parameter and method requested
- Furnish the Program Manager with the laboratory procedures specified in the QAP
- Review the FSP to assure that the samples will provide sufficient material for the specified analyses and the sample preservation and holding times can be met
- Perform the analyses and provide the Program Manager with the results and the specified **Quality Assurance/Quality Control (QA/QC)** documentation
- Support the activity NAVOSH Program and participate in the risk management process to ensure site specific safety and health hazards are identified and appropriate controls applied where needed (Supervisors must apply an appropriate level of supervision to ensure the risk management process is effective.)

3.2.4 Health and Safety Personnel. Facility Health and Safety Personnel must:

- Coordinate with the Program Manager, laboratory and sampling personnel to ensure an appropriate site specific risk management process is completed in addition to maintaining a functional NAVOSH program for DOD environmental compliance sampling and laboratory personnel
- Determine when a site specific "Health and Safety Plan", as required by 29 CFR 1910.120, is necessary (When it is, ensure that **Appendix C**, Health and Safety Plan Review checklist is used to make certain the plan meets all requirements whether generated in-house or by contract. Contact the Navy Environmental Health Center, Environmental Programs Directorate to make sure **Appendix C**, Health and Safety Plan checklist is the most current version available.)
- Assist in the preparation of a "Health and Safety Plan" should one be required and generated in-house
- Review **Standard Operating Procedures (SOPs)** to ensure safety hazards and precautions are adequately addressed
- Furnish trained personnel to perform industrial hygiene monitoring and health and safety oversight and support during field sampling as specified in the safety plan or HASP

3.3 CONTENTS OF SAMPLING AND ANALYSIS PLAN. The **Sampling and Analysis Plan (SAP)** should be prepared based on the regulatory requirements, the information required for making decisions, qualifying factors of the sampling and laboratory operations, and OPNAVINST 5090.1B Chapter 25. The SAP contains the documented quality system requirements appropriate to meet the type, range, and scope of the sampling and testing being performed. The **SAP** consists of:

- A detailed **Quality Assurance Plan (QAP)** addressing regulatory program requirements
- A **Field Sampling Plan (FSP)**, extracted from the QAP, consisting of only that information required by sampling personnel
- A site specific **Health and Safety Plan (HASP)**, meeting the requirements of **Appendix C**, when the scope of work requires it [See 29 CFR 1910.120(a)(1)(ii) or (iii) to determine applicability.] (For many or most compliance sampling events, a less extensive and less detailed **Site Specific Safety Plan** will be included. Such plans, which do not need to meet all the criteria of 29 CFR 1910.120, must still reflect the five step risk management process detailed in **Section 3.3.3.1** and include minimum elements as outlined in **Section 3.3.3.2**.)
- A **Quality Assurance Manual (QAM)** for all testing operations documenting the quality system followed by the laboratory or field testing operation (The QAM is reviewed to ensure that requirements of the QAP are part of the laboratory operation. The contents and preparation of the QAM are beyond the scope of this manual.)

NOTE:

The field sampler is a vital component of a Sampling and Analysis Plan. The sampler's participation and understanding of the requirements will contribute to the success of the program.

3.3.1 Quality Assurance Plan (QAP).

3.3.1.1 Description. The Quality Assurance Plan is prepared in accordance with EPA instructions. The QAP defines the regulatory program objectives for assessing environmental data and specifies the samples, sampling and analytical methods, documentation, chain of custody procedures, and quality assurance requirements. The QAP provides guidance and direction for personnel to follow. The extent of detail in the QAP is based on the requirements of the compliance program. All aspects of compliance should be detailed in the QAP to ensure consistent execution of the data collection activities. Changes made to the QAP provide a documented audit trail.

3.3.1.2 QAP Elements. The elements to consider when preparing a QAP are items included in a documented quality system. The elements contained in the QAP may refer to other documents, procedures, or methods and may provide the listing of the documentation references for the project. The elements presented here are a comprehensive list that may be used by the Program Manager as a checklist when preparing to meet compliance requirements. Not all the elements are required for all compliance programs.

The QAP should consist of the following elements as needed for the project:

I. TITLE PAGE

- Title of document (Quality Assurance Plan)
- Sampling program name
- Job number
- Date
- Revision number
- Name, organization, and phone number of Program Manager
- Approval Signatures

II. TABLE OF CONTENTS

III. PROJECT DESCRIPTION

- Provide a comprehensive review of the data gathered during previous studies to define the chemicals expected to be present, if applicable.
- Identify the contaminants of concern for this project.
- Present the project objectives, including the decision criteria applicable to each parameter analyzed, based on regulatory compliance standards.
- Provide the sample network design and rationale and describe how the work will meet these objectives.
- Prepare a table that summarizes the matrices to be sampled, the parameters to be analyzed in the field and laboratory, and the sampling frequency. This table should also be included in the FSP.
- Identify the intended data uses and determine the **Data Quality Objectives (DQOs)**. It is important that the DQOs be determined at the start of the project so that the data collection program can be designed to this end. In most cases, the DQOs are already established as part of the compliance program specified by law or regulation.
- Provide an implementation schedule.

IV. PROJECT ORGANIZATION

- Define the responsibilities for ensuring the collection and assessment of valid measurements and data. The individuals assigned responsibility for the following activities should be identified by name, organization and phone number:
    - Program Manager - overall project manager and person responsible for use of the data
    - Field operations
    - Field quality control
    - Laboratory analyses
    - Laboratory quality control
    - Data validation
    - Review of sampling and testing protocols to meet specific regulatory requirements
    - Review of Laboratory and Field Testing Performance Evaluation Sample results
    - Systems auditing
    - QA/QC Coordinator
    - Overall Safety and Health Coordinator
- Show the project organization in an organization chart. Include the roles and identity of key organizations and individuals.
- Assemble the Sampling and Analysis Team(s). Collection, field testing, and documentation of valid environmental samples depends on correct planning and the competence of the personnel performing the field work. Field sampling personnel must be trained in the requirements for documentation and the specific procedures to be used to collect the samples. In addition, health and safety training may be required to ensure that field sampling personnel are able to identify hazardous conditions and to properly use the specified **Personal Protective Equipment (PPE)**. Medical surveillance of field sampling personnel may be required to ensure that they are physically capable of using the personal protective equipment and that they do not have health problems which would make them more susceptible to injury from accidental exposure to hazardous chemicals.
- Consideration must be given during preliminary planning to the possibility of encountering energetic materials during sampling. If there is such a possibility, expert assistance should be obtained (i.e., Explosive Ordnance Disposal)

during development of the FSP and safety plan or HASP as appropriate.

- Staff the sampling team with the necessary number of trained personnel to perform the sampling actions required. The Program Manager is responsible for ensuring that functions are properly staffed. Sampling programs in hazardous or unknown hazard locations may require complex functions to be assigned to specially trained team members, by the Program Manager.

NOTE:

For small sampling programs, such as collecting drinking water samples or wastewater compliance samples, the Program Manager may perform all of the functions.

- Train all personnel who contribute to the development of the SAP or who collect or analyze samples. Proper training for the functions they perform must be documented. For a listing of training sources, refer to **Appendix B**. Records shall be maintained, and updated as necessary, to document each individual's training, skills, experience, and qualifications.
- Describe the training of personnel who perform any of the functions of the **Core Team**. Formal training in the operations to be performed must at least include:
    - Minimum training requirements of OPNAVINST 5090.1B, 25-5.8, for Program Manager, Sampling Personnel and Laboratory Personnel
    - Basic sampling techniques (grab sampling, composite sampling, how to avoid contamination, use of preservatives, etc.)
    - Specific sampling techniques as required (e.g., National Pollution Discharge Elimination System (NPDES) sampling, potable water bacteriological sampling)
    - Completion of environmental sampling paperwork including **Sample Container Labeling, Field Logbooks, Field Sampling Forms, and Chain of Custody (COC) Records**
    - Environmental laws and regulations
    - Field preservation techniques
    - Field testing techniques
- Document safety and pre-assignment training of all team members prior to site arrival. Copies of the documentation shall be provided to the Program Manager. Specific but not all inclusive safety training requirements shall include as needed:
    - Hazard Communication training, 29 CFR 1910.1200(h)
    - Hazardous Waste site worker/emergency response training, 29 CFR 1910.120
    - Confined space entry training, 29 CFR 1910.146(g)
    - Mandatory training for all team members specifically required for Treatment, Storage and Disposal and clean-up operations are identified in 29 CFR 1910.120(e)
    - Additional safety training requirements for the use of respirators, protective clothing, weight and material handling, etc. are identified in OPNAVINST 5100.23 (series) and local safety instructions
- Describe the communication procedures for the project and notification routes

Team function responsibilities may include:

CORE TEAM:

Program Manager:

- Ensures adherence to **Sampling and Analysis Plan (SAP)**
- Ensures that required personnel are available to perform the work
- Trains Field Sampling Team to ensure each member understands the program goals and their individual responsibilities
- Coordinates schedules with facility personnel to ensure access to sampling locations
- Coordinates with facility health and safety staff, where relevant
- Oversees sampling, including:
    - Collection of samples at the specified locations
    - Preparation of the specified number of sample containers for each sample, including QC samples
    - Adherence to sample preservation require-ments
    - Field testing
    - Coordination with laboratory
- Maintains the **Field Log Book** or **Field Note(s)**

Field Sample Custodian:

- Labels and packs the sample containers
- Completes the **Field Log Book/Field Notes**
- Prepares and completes the **COC Record**
- Delivers/ships the samples to the laboratory
- Provides for physical security for samples to ensure that no tampering has occurred, when required by the QAP

Field Sampler:

- Operates sampling equipment

- Records observations of site conditions and relevant field data, such as discharge flows, water levels and other equipment readings
- Collects samples in containers specified in the SAP
- Preserves samples (filtration and/or addition of preservatives) as required in the SAP
- Maintains personal certification as required by some state agencies and regulatory programs prior to sampling

Field Chemist:

- Performs specified field tests
- Calibrates field test instrumentation
- Documents observations
- Follows Quality Assurance requirements
-

Equipment Technician:

- Procures and assembles required equipment and sample containers
- Moves equipment and unused sample containers to the site and between sampling locations
- Decontaminates sampling equipment between samples, as needed
- Assists the Field Sampler in using the sampling equipment as required

QA/QC Coordinator:

- Conducts and documents an internal audit on a continual basis at least once per year
- Monitors operations to ensure equipment, personnel, activities, procedures, and documentation conform with the quality system established

Health and Safety Supervisor:

- Assures compliance with Health and Safety Plan.
- Calibrates personnel air monitoring instruments and records data
- Performs real-time personnel air monitoring as required to assure that proper respiratory protection is being used
- Establishes the boundary of the **Exclusion Zone** and **Contamination Reduction Zone** based on weather conditions and the results of air monitoring, as needed
- Collects industrial hygiene monitoring samples as required to document exposure of sampling team to toxic chemicals
- Obtains **Confined Space Entry Permits** from the facility **Confined Space Program Manager**, or issues the Confined Space Entry Permit if the site is not covered by a Confined Space Entry Program
- Coordinates with the facility **Certified Respiratory Protection Program Manager** as needed on the requirements for use of respirators during sampling activities
- Coordinates with the facility to ensure the availability of emergency response services (Fire, Ambulance, Emergency Management Team)
- Establishes communication procedures for obtaining emergency response support
- Supervises emergency response activities
- Coordinates with the Program Manager as required to modify the HASP to reflect new or changed conditions

SPECIALTY PERSONNEL:

Survey Team:

- Locates and documents sampling locations with respect to a permanent marker(s)
- After initial groundwater well installation, establishes the elevation of the top of the casing of the sampling wells for future comparison (Future well depth and water level readings for routine monitoring may be performed by trained samplers.)

Geologist:

- For soil sampling surveys, classifies samples by soil type and documents changes in soil type as a function of depth below surface
- During installation of groundwater monitoring wells, examines well cuttings to define subsurface soil types and geology

Well Driller:

- Operates drill rig for deep soil sampling and installation of groundwater monitoring wells
- Installs groundwater monitoring wells

Security Officer:

- Marks **Exclusion Zone and Contamination Reduction Zone** boundaries
- Patrols **Contamination Reduction Zone** boun-dary to prevent unauthorized access
- Guards equipment, decontamination facilities, etc., as required to ensure materials are not stolen, damaged, or tampered with

PPE/Respirator Technician:

- Sets up decontamination facility
- Assists other personnel with personal protective equipment
- Inspects and decontaminates respirators
- Assists in decontaminating personnel
- Supervises supplied air equipment or supply of compressed air cylinders

Safety Observer:

- Observes and communicates with personnel working in confined spaces (has no other duties during confined space entry)
- During work requiring air respirators (not Confined Space), remains outside Exclusion Zone but dressed in PPE for immediate emergency response and maintains communication with personnel in Exclusion Zone

Laborers and Equipment Operators:

- Move drums to sampling areas
- Operate drum opening equipment
- Dig soil sampling access pits
- Package and dispose of wastes generated during sampling

V. QUALITY ASSURANCE OBJECTIVES FOR COMPLIANCE DATA

- Describe the documents and references used in preparing the QAP.
- Present the Quality Assurance Objectives for the Project, Field, Laboratory, Computer and any other activities associated with the project.
- Identify the extent and need for legally defensible procedures for all sample and data collection activities. The specified methods include sample handling requirements, method detection limits, and precision and accuracy of the measurement data. The use of the approved sampling and testing method ensures representativeness and comparability to the regulatory standards. Alternative or non-standard methods require more elaborate DQO statements.
- Define the levels of quality required for the data. The data quality objectives should be based on an understanding of the intended use of the data, the measurement process, and the availability of resources.
- Express the data quality in terms of precision, accuracy, completeness, representativeness, and comparability. As a minimum, requirements should be specified for detection limits, precision, and accuracy for all types of measurements.
    - **Precision** is an expression of the degree of reproducibility of results, or the degree of mutual agreement among independent, similar, or repeated measurements. Precision is monitored through the use of replicate samples or measurements, and is reported as a standard deviation, standard error, or relative standard deviation. Multiple replicate samples normally are taken to assess precision in field sampling.
    - **Accuracy** is the degree of agreement between a measured value and the true value. It may be monitored in a program through the use of blank samples or standard reference materials. For field quality control, samples are routinely spiked with a known reference material. In an analytical laboratory, accuracy generally is expressed in terms of percent recovery of a standard. Examples of ways to help meet accuracy goals include standard methodology, performance audits, traceability of instrumen-tation, traceability of standards, traceability of samples, and referenced or spiked samples.
    - **Completeness** may be evaluated by carefully comparing project objectiveness with proposed data acquisition. Completeness is the amount of data collected compared to the amount of data expected or required under ideal conditions. One expression of completeness would be the percentage of collected samples that was completely analyzed or the percentage of data points actually required compared to those planned to be acquired. Completeness may represent the quantity of data that must be acquired to meet project needs, as well as the percent recovery required to ensure data adequacy.
    - **Representativeness** refers to 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. The acquisition of a representative sample may be based on statistical sampling that dictates the approach, number, conditions, and even location of samples to be collected or analyzed. The representativeness is a quality characteristic that attempts to define how a sample will be collected to ensure its relationship to the media being sampled. For most water monitoring studies, it should be considered a goal to be achieved rather than a characteristic that can be described in quantitative terms.
    - **Comparability** must be assured in terms of sampling plans, analytical methodology, quality control, data reporting, and similar essential factors. Comparability is the degree to which data from one study can be compared to other, similar studies. Such comparisons are facilitated when internally consistent measures are used throughout the effort. Important examples of data comparability are standardized citing, sampling, and analyses; reporting units consistently; and standardized data format. Strict adherence to standardized methods and protocols when conducting a test, along with use of performance evaluation samples and referenced materials, alleviates many of the

comparability concerns that otherwise may occur.

VI. FIELD INVESTIGATION SAMPLING PROCEDURES

- Describe, in detail, the rationale and location of the samples to be collected.
- Reference the specific regulatory program used for guidance. Reference the sampling methods used for developing the field sampling activities.
- Explain in detail the sampling activities needed for the FSP.
- Explain in detail the use of alternative methods along with the rationale for using sampling methods from other regulatory programs or alternative sources.
- Document sampling procedures and field activities exactly as performed. Deviations to documented procedures are noted during the sampling or field operation.
- Describe sample containers, sample volume, sample type, quality control samples, sample storage, equipment selections, reagent quality, supplies, preservation, holding times, decontamination, waste disposal, sample packaging and shipping.

VII. SAMPLE AND DOCUMENT CUSTODY PROCEDURES

- Define the custody procedures including document custody, document control, laboratory custody, and compliance files. The field custody procedures may be referenced at this point and summarized, but should be described in detail in the FSP. The degree of custody documentation is based on the data gathering needs. Legal chain of custody is not always required in compliance monitoring. The QAP must clearly state the degree of custody required for routine compliance monitoring.
- Identify and discuss the following elements of the custody procedures:
    - ***Project Requirements***
        - Custody procedures for compliance gathering files, if required
        - Maintenance of document control system
        - Proper assignment of unique sample identification numbers
    - ***Field Procedures***
        - Procedures for recording exact location of each sample, sample labeling, Field Log Books/Field Notes
        - Use of sample tracking, custody transfer and COC Records in the field
        - Use of sample custody seals (Optional)
        - Specification of procedures for sample handling, storage, and shipment
    - ***Laboratory Procedures***
        - Identification of Sample Custodian
        - Procedures for sample scheduling, manage-ment, receipt, handling and disposal
        - Use of laboratory custody forms and sample tracking documentation requirements within laboratory operation (Describe need for internal laboratory documented legal custody.)
- Specify in the custody procedure the documents that must be maintained and the responsibility for retaining the records that comprise the final compliance files. The document tracking system must be described in the QAP.
- Describe the process for traceability of all entries in Field Logbook(s)/Field Notes, laboratory form(s) and computer record(s) to ensure tracking to the chain of custody or other designated project control document(s).

VIII. CALIBRATION PROCEDURES AND FREQUENCY

- Describe the calibration and frequency of calibration or standardization of both field and laboratory instruments.
    - For field instruments, the manufacturers' calibration and maintenance procedures should be incorporated into the FSP. The **FSP** should include step-by-step calibration procedures, frequency, equipment maintenance logs, instrument accuracy criteria, standards traceability, records, corrective action procedures, and equipment limitations.
    - For laboratory equipment, the calibration procedures may be quite extensive because of the number and variety of instruments used for performing analyses. Therefore the laboratory methods should be referenced and summarized in the QAP. The requirements for instrument calibration and frequency are specified by the EPA in the regulatory method. The quality control requirements for the analyses performed should be specified in the QAP.

IX. ANALYTICAL PROCEDURES

- Select the analytical procedures at the start of the project based on regulatory program requirements and the ability of the procedure to produce data in the matrix being tested. An initial demonstration of method performance in the matrix may be conducted to ensure the data quality objectives are achievable.

- Specify the analytical procedures to be used that meet the regulatory program requirements. Standard analytical methods (e.g., *American Society for Testing and Materials, Standard Methods for the Examination of Water and Wastewater, 18th edition*) may be included by reference to the method number and laboratory procedure SOP. Non-standard methods should be prepared as SOPs and included with the QAP.
- Specify the required reports to be submitted by the testing operation, including data formats, units of measure and required quality control documentation.
- Describe the process for modification and changes to the analytical procedures including documentation and control to ensure consistency of the data. Data trends may be affected by procedure modifications.
- Select the laboratory based on a review of the certification or accreditation requirements of the regulatory program. As required by OPNAVINST 5090.1B, all testing should be performed by laboratories having appropriate credentials. Credentials may be required for the specific type of regulatory testing, such as the **Safe Drinking Water Act (SDWA)**, and for the specific test and parameter. Certification or accreditation will list approved test methods, matrix or regulatory program in the scope of accreditation. Certification by the state, territory or EPA where the drinking water supply is located is required under SDWA. **Clean Air Act (CAA), Clean Water Act (CWA), Resource Conservation and Recovery Act (RCRA)**, and **Toxic Substance and Control Act (TSCA)** programs are state specific to the need for certification prior to performing testing and sampling.
- Demonstrate that the laboratories meet the accreditation requirements prior to performing work for the Navy. The scope of accreditation must be reviewed and documentation provided prior to selecting the laboratory for performing the required testing. Accreditation requirements apply to fixed, mobile and field laboratories. Accreditation programs include laboratory site assessments, requirements for quality control data, and participation in on-going proficiency testing. Each Navy laboratory should be certified or accredited by a Federal, state, or a third party nationally recognized accreditation system for all testing performed by the laboratory.

X. INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

- Present laboratory and field quality control checks separately. Specific methods may be described or included by reference. The reference method describes the laboratory QC and may include suggested field QC.
- Present the field QC checks including the procedures and frequency for:
  - ° Field blanks
  - ° Field duplicates
  - ° Trip blanks
  - ° Matrix spike/matrix spike duplicates
  - ° Equipment decontamination blanks
- Present the laboratory QC procedures used by each laboratory performing analyses for the project. Procedures and frequency are to be included for:
  - ° Laboratory duplicates
  - ° Method blanks
  - ° Method spikes
  - ° Calibration standards and continuing calibration
  - ° Internal standards
  - ° Surrogate spikes/recovery
  - ° Reference samples or QC Check samples
  - ° Matrix spike/matrix spike duplicates
- Specify the use and frequency of field and laboratory control charts and control criteria.

XI. DATA REDUCTION, VERIFICATION, RETENTION AND REPORTING

- Specify the field, laboratory and computer QC procedures used for data reduction, verification and reporting. Equations and computational procedures should be documented. The procedures of each laboratory performing analyses must be included or referenced to compliance standards. Data reporting formats should be specified in the QAP.
- Validate completed data packages at the laboratory against criteria specified by reference to standard method or as documented in the QAP. The need for third party or independent validation is not required for most compliance programs.
- Specify the time and manner of records retention and retrievability. Raw and final data must be retained as required by the regulation, contract requirement or for at least three years. Records must be retrievable within a reasonable time while in storage.

XII. PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

- Conduct routine performance audit of each process on a continual basis to assure adherence to the SAP. Process audits or Performance audits are quantitative, systematic checks on the quality of an operation. Performance audits may be completed in phases or by operation. Performance audits verify that SOPs are being followed and fully implemented. In contrast, system audits are qualitative reviews of the entire data production process and include on-site evaluations of field operations and laboratories. System audits include both internal and external audits.

- Present a discussion of the nature and frequency of internal audits of field and laboratory operations. The QA/QC Coordinator is responsible for performing the audit following the procedures included in the QAP.
- Conduct and document internal audits on a continual basis at least once per year.
- Conduct external audits by an auditor independent of the organization that is audited. The Environmental Compliance Evaluation (ECE) second echelon review and the Installation Restoration Laboratory Evaluation Program review shall be considered the external review of the operations.
- Detail and record all investigations of subcontractors proficiency and conformance to the SAP requirements. Subcontracting of any sampling or testing functions must adhere to the same requirements and audits as non-contracted operations. Subcontracting must be approved by the Program Manager and must include documentation that all quality system program requirements for the project are satisfied.

XIII. PREVENTATIVE MAINTENANCE PROCEDURES AND SCHEDULES

- Specify the **Preventative Maintenance (PM)** procedures and schedules for field, laboratory and computer equipment.
- Perform PM according to the manufacturer's procedures and schedule for all the field instruments and equipment. Reference to the appropriate SOP's must be listed and copies must be available for review.
- Present the PM procedure of the laboratory by reference to laboratory SOPs or QAM. The laboratory instrument PM procedures and frequency may be elaborate and complex. Procedures and documentation of all PM and maintenance must be available for review.
- Label, mark or otherwise indicate the working status of all equipment and instruments used for measuring, to ensure non-functioning equipment is not used for measurement.

XIV. EVALUATION OF SAMPLING/ MEASUREMENT PROTOCOLS

- Describe the acceptance criteria for field and laboratory data along with the regulatory standard for compliance.

NOTE:

In the drinking water program the acceptance criteria for laboratory performance is found for each parameter in the Code of Federal Regulations and referenced methods. The regulatory standard (such as the Maximum Contaminant Level or Maximum Contaminant Level Goal) for each parameter is given in the regulation.

- Describe the actions to be taken when evaluating the field, laboratory and regulatory criteria.
- Present the procedures for data usability when sample collection deviations occur in the field, test procedures are modified due to matrix effects and handling of other events that may not be known at the start of the project.

XV. CORRECTIVE ACTION

- Present corrective action procedures including:
    - Define a mechanism to set data acceptability limits
    - Identify defects in sampling and analysis
    - Trace defects to their source and record the total number found
    - Plan and implement measures to correct defects
    - Maintain documentation of the results of corrective action
    - Continue using the process until each defect is eliminated or minimized
- Define corrective action procedures for laboratory and field analyses. The laboratories and field operations should summarize in the QAP the elements of their SOPs that deal with the previously described corrective action mechanism.
- Present procedures for the resolution of complaints or circumstances raising doubt concerning the sampling and testing processes. Records shall be made of the complaint or circumstance and the resolution of all complaints.

XVI. QUALITY ASSURANCE REPORTS TO MANAGEMENT

- Present a schedule for the periodic reporting of measurement system performance to the Program Manager. The QA reports must contain information on any event, or the absence thereof, that results in a variance to specified procedures or a reduction of quality below that specified in the Data Quality Objectives. Reports should include:

- ° A description of any variance, discrepancy or problem
- ° The implications of the same on DQOs
- ° The required corrective action and criteria that must be met to achieve the site DQOs
- ° A schedule for completing the corrective action

3.3.2 Field Sampling Plan (FSP).

3.3.2.1 Description. The FSP contains the information required in the field to properly perform field tests and collect samples for testing. It is important that the FSP be consistent with the QAP. The information in the FSP is taken from the QAP and specific detail added for the field operations. In very limited sampling and testing programs the QAP, FSP, QAM, and HASP may be the same document. The FSP provides specific direction for the sampler. Detailed procedures are specified as to the sampling locations, frequency, equipment and quality assurance items. Health and safety hazards are also provided.

3.3.2.2 FSP Elements. The following elements may be used as a **checklist** by the sampler when preparing for any sampling and testing operation. The amount of detail and the inclusion of any section is dependent on the data quality needs of the project. Not all of the following elements may be required for all compliance sampling events. Any SOPs meeting the requirements of the regulatory program may be incorporated by reference through the QAP or FSP.

I. TITLE PAGE

- Title of document (Field Sampling Plan)
- Sampling program name
- Job number
- Date
- Revision number
- Name, organization, and phone number of Program Manager

II. TABLE OF CONTENTS

III. INTRODUCTION

- Explain where and why the samples are being collected such as:
  - ° Permit requirement
  - ° Determine the drinking water quality
  - ° Characterization of waste material for disposal
- Identify the responsible individuals, by title, name and telephone number for Program Manager, QA/QC Coordinator, HASP Officer, Sampling Operations Manager, Laboratory Manager (however named) and other members of the project team.

IV. OBJECTIVES

- Present the project objectives, including the decision criteria applicable to each parameter analyzed.
- Describe how the work will meet these objectives.
- Explain the sampling operation as to the number of personnel, education, training, technical knowledge and experience needed to meet assigned functions.

V. SAMPLE TYPES

- List sample matrix (soil, ground water, surface water, etc.).
- Present sampling method (grab, composite, continuous).
- Present field tests and field measurements required.

VI. CHEMICAL CONTAMINANTS OF INTEREST

- Present a table showing contaminant versus matrix, or listing contaminants if only one matrix is to be sampled.

VII. HEALTH AND SAFETY HAZARDS

- A site specific Health and Safety Plan (HASP), meeting the requirements of **Appendix C**, will be included when the scope of work requires it. See 29 CFR 1910.120(a)(1)(ii) or (iii) to determine applicability.
- For many or most compliance sampling events, a less extensive and less detailed site specific safety plan will be included. Such plans, which do not need to meet all the criteria of 29 CFR 1910.120, must still reflect the five step risk management process detailed in **Section 3.3.3.1** and include the minimum elements outlined in **Section 3.3.3.2**. At a minimum, the plan should describe the specific expected hazards, safe operating procedures, Personal Protective Equipment (PPE), and training required to collect the samples in a safe manner. PPE recommendations must be specific as to type and include any special decontamination or disposal requirements which apply. Include the warning indications and health effects of each significant toxic contaminant (include a Material Safety Data Sheet (MSDS) in the FSP, for each chemical).

VIII. SAMPLING LOCATIONS AND FREQUENCY, INCLUDING QUALITY CONTROL SAMPLES

- Describe or reference detailed sampling location, including a map or sketch as relevant. Assign a unique code number or description to each location (this may be X and Y coordinates for a sampling grid or the outfall number from the permit). For soil, waste pile and surface water samples, include sampling depth(s) at each location. For drinking water include the exact location so that any person returning to the site will be able to locate the specific tap sampled.

- List required frequency of each type of quality control samples, including:
  - Field duplicates
  - Matrix spike/matrix spike duplicates
  - Field blanks
  - Trip blanks
  - Equipment decontamination blanks
- List required time of sampling if relevant (immediately after a rain event for surface runoff contaminant samples).
- List frequency of sampling if periodic samples are required (monthly, quarterly, annually) as specified by permit or regulatory requirements.
- List type of sample collected such as grab, flow proportional, or time composites.
- Monitor, control and record all environmental conditions that may invalidate the results or adversely affect the representativeness of the sample.

IX. GENERAL SAMPLING INFORMATION

- Describe for each type of sample:
  - Sample Containers (number, size, type, material, cleanliness requirements)
  - Sample Volumes (minimum quantity required per container)
  - Preservation Techniques
  - Holding Times
  - Number and Type of Field QC Samples
  - Equipment Decontamination Blanks
  - Field Duplicates
  - Matrix Spikes
  - Trip Blanks
  - Split Samples
- Describe the monitoring, controls and recording of environmental conditions, as appropriate, to ensure that the results are not invalidated due to the site conditions. Items to be controlled and monitored include but are not limited to: weather, climate, and background or area contamination.

X. SAMPLING EQUIPMENT, REFERENCE MATERIALS AND SUPPLIES

- Label, mark or otherwise identify all equipment, instruments, reference materials and associated supplies to indicate the calibration or standardization status.
- Describe the procedures for measurement traceability to national or international standards where applicable.
- Describe the procedures for labeling, documenting preparation, and use of reagents and solutions. Labeling should include identity, concentration, grade, and quality of the material. Documentation of preparation and traceability to stock material should be described in detail.
- Specify a complete list and estimated quantities of materials and supplies required to perform the sampling, including:
  - Equipment required for field measurements (thermometer, pH meter, etc.)
  - Sample containers (number of each size, type and material)
  - Preservatives, including pH paper and pipets as appropriate
  - Sampling equipment, including screens, filters, bailers, etc. as appropriate
  - Field Log Book/Field Notes
  - Waterproof pen
  - COC Records (prenumbered)
  - Three part sample container labels
  - Plastic tape to cover container labels, seal shipping containers, etc.
  - Sample shipping containers
  - Packaging material (type and quantity as appropriate)
  - Plastic bubble wrap
  - Vermiculite
  - Resealable bags
  - Metal paint cans
  - Custody Seals (Optional)
  - Resealable bags for **COC** Records
  - Ice in sealed plastic containers or resealable bags
  - Equipment and supplies for decontaminating sampling equipment
  - Containers for contaminated waste material generated during sampling (purge water, used preservative, disposable PPE, etc.)
  - Personal protective equipment and respirators (See Health and Safety Plan)
  - Record all maintenance and perform all preventative maintenance per the operating procedures or manufacturer's instructions.

XI. SAMPLING PROCEDURES

- Specify in step by step procedures, exactly how the samples are to be collected and any field descriptions or analyses that are required. Cover in detail or reference standard procedures for:
  - Field Operations
    - Soil: location (survey requirements), color and soil type by depth increment

        - Liquid: viscosity, color, opacity (cloudiness) homogeneity, flow rate or quantity per container
        - Solid wastes: description (type, color, homogeneity; quantity per pile or container
        - Groundwater: well installation requirements, purging requirements, pH, temperature, conductivity, depth to groundwater
        - Air: weather conditions (rain, fog, humidity, temperature, wind velocity); visible par-ticulates
        - Stack gas: specify parameters and procedures
    - Sample Collection: Complete a **Field Log Book/Field Notes** describing the samples collected and sample containers filled at each location. Reference to the sample collection procedure reduces the amount of field time. Record is made as to the procedure followed anddeviations, modifications, or variations noted. Include copies of the example Field Log Book pages/Field Notes as an attachment to the FSP. The procedure or field notes must describe in detail:
        - How the material for each sample container is collected (grab, composite, continuous for specified period, etc.)
        - How each sample is treated (screened, filtered, sequence for filling ground water sample containers, etc.)
        - Preservatives for each sample container (type, procedure for using)
        - Procedure for preparing each type of quality control sample
    - Field Decontamination Procedures
        - Sampling Equipment
        - Sample containers used for field testing
    - Waste Disposal Procedure
        - RCRA requirements
        - Navy or Site specific requirements
        - Samples, decontamination materials, equip-ment and all field activities
- Record the following information for all sampling operations:
    - Sampling date
    - Sampling time
    - Locations sampled, with tables, graphs, sketches, and photographs as appropriate
    - Name of individual(s) collecting the samples
    - Unambiguous identification of sample
    - Type of sample
    - Description of sample
    - Reference to sample collection procedures
    - Preservation used
    - COC Documentation
    - Measurements, examinations and results
    - Calibration or standardization of equipment
- Specify the reporting procedure for each sampling event. Data must be reported accurately, clearly, unambiguously and objectively within the guidance of the instructions for the operations. Reports may be in the form of field forms, copies of logbook pages or formal consolidated reports describing the operation or event. Reports must include at a minimum:
    - The identification of the operation
    - Subcontractor(s)
    - Location of sampling
    - Unique identification of the report
    - Page numbers
    - Name and address of customer
    - Description and unambiguous identification of the sample
    - Characterization and condition of the sample
    - Unambiguous description of non-standard method
    - Reference to sampling procedure
    - Reported measurements with units of measure
    - Signature and title of person accepting responsibility for the content of the report
    - If amended, the report must be identified and the changes documented
- The Sampler or Program Manager must notify all affected parties, in writing, of any event such as identification of defective measuring devices or equipment that casts doubt on the validity of the sample collection process or results given in the report.
- The HASP should provide information to emergency medical care service(s) at a convenient and readily accessible medical facility and establish emergency communications with emergency response services. Personnel injured as a result of an accident at the site should be handled in the following manner:
    - First aid equipment should be available on site for minor injuries.
    - The injured employee should be transported by local emergency vehicle to the appropriate medical facility.
    - Written report of accident should be prepared by the program administrator within 48 hours.

XII. SAMPLE CUSTODY AND DOCUMENTATION

- Record and report all sampling data with sufficient figures to be statistically significant.
- Describe the calibration, standardization, measurement and custody records and the retention period. The records gathered during monitoring or

sampling must be the same period of time as field and analytical data.

- Present procedures for reviewing field records for accurate reporting and adherence to documented procedures. Procedures should include the mechanism and approval required for modifying or amending records and data.
- Specify in detail the procedures to be used to identify each sample and to document the collection procedures, field test results, and chain of custody for the laboratory samples. The type of custody to be used is dependent on the sampling and analysis required and will be specified in the QAP. If data is known to be used in litigation prior to sampling, legal chain of custody procedures must be followed. Include information on:
  - ° Sample Identification Numbers
  - ° Field Log Book/Field Notes
  - ° Field Custody Procedures
  - ° Sample Container Labels
  - ° Custody Seals (Optional)
  - ° COC Record. Where appropriate, include a copy of the laboratory's COC Record for legal tracking of the sample(s).
  - ° Custody Transfer Procedures (Optional)

XIII. SAMPLE PACKAGING AND SHIPMENT PROCEDURES

- Specify any special procedures that are to be used to pack the sample containers for shipment.
- Specify how often samples are to be shipped (daily, all at once, etc.).
- Specify limitations on holding times for the samples.
- Specify how the samples are to be shipped.
- Specify the manifest requirements for sample containers that are shipped to a laboratory. Include:
  - ° Specific name that is to be used to describe the material
  - ° The hazard class that is to be specified on the manifest for each shipping container
  - ° If the samples are classified as hazardous, list the 24 hour notification number that is required on the manifest
- Record storage conditions, and variations to documented sample handling, preparation, shipping and storage procedures.

XIV. LABORATORY

- Specify where the sample containers are to be sent for analysis. For each laboratory, list:
  - ° Laboratory name
  - ° Address
  - ° Name of notification person
  - ° Phone number
  - ° Notification process and procedure

3.3.3 Navy Occupational Safety and Health (NAVOSH) Program.

3.3.3.1 Description. OPNAVINST 5100.23D, *Navy Occupational Safety and Health (NAVOSH) Program Manual*, is the primary reference document describing the NAVOSH Program. All elements of this manual apply to protecting the health and safety of Navy DOD environmental compliance personnel. The relationship between OSHA standards and the NAVY Occupational Safety and Health Program is detailed in Chapter 16 of OPNAVINST 5100.23D. OPNAV instructions, particularly the contents of OPNAVINST 5100.23D and any instructions issued by the command having specific technical cognizance or assigned responsibility in OPNAVINST 5100.8G and approved by the Chief of Naval Operations, are considered to be NAVOSH standards which govern the NAVOSH Program.

3.3.3.1.1 Site-Specific Safety and Health Concerns. The NAVOSH Program should not be considered all inclusive since the hazards and environments encountered during compliance sampling are so diverse. A partnership must exist between compliance sampling personnel and cognizant industrial hygiene and safety professionals to ensure site specific safety and health hazards are identified and addressed.

Section 3.2 identifies specific responsibilities. A site specific Health and Safety Plan (HASP) meeting the requirements of **Appendix C** will be included when the scope of work requires it, per 29 CFR 1910.120(a)(1)(ii) or (iii). A much less extensive/detailed site specific safety plan will be included for the usual compliance type sampling which does not fall under 29 CFR 1910.120. This safety plan should reflect the five step risk management process detailed below. At a minimum, the plan should describe the specific expected hazards, safe operating procedures, Personal Protective Equipment (PPE), and training required to collect the samples in a safe manner. PPE recommendations must be specific as to type and include any special decontamination or disposal requirements which apply. The warning indications and health effects of each significant toxic contaminant (include a Material Safety Data Sheet (MSDS) in the FSP, for each chemical) should be included.

3.3.3.1.2 Risk Management Process. Risk management is a five step process: Identifying Hazards, Assessing Hazards, Making Risk Decisions, Implementing Controls, and Supervising. It is a systematic way of thinking, used to better assess and control risk. Identifying the hazards involves a preliminary hazard analysis where the hazards and possible causes of hazards are listed. Assessing the hazards involves prioritizing the identified hazards by severity of possible loss and probability of possible loss. Making risk decisions involves considering all available risk control options, starting with the most serious risk first and referring to the list of possible causes developed during the preliminary hazard analysis. You must then determine whether the benefit outweighs the risk and, when appropriate, contact higher authority for guidance. Control of hazards may be accomplished through engineering and/or administrative controls as well as Personal Protective Equipment (PPE). An appropriate level of supervision to monitor for effectiveness of controls and watch for changes is critical.

The risk management process has to occur at all levels to be effective. As previously stated, a partnership must exist between compliance sampling personnel and cognizant industrial hygiene and safety professionals to ensure site specific safety and health hazards are identified and addressed. The best safety plan devised for a particular site can be rendered useless if changing conditions are not monitored by the individual performing the work and taken into account for their personal risk assessment.

3.3.3.1.3 Risk Management Example. For illustrative purposes, a limited example of the risk management process is provided as follows: The scenario is sample collection at a dammed lake and its' outfall. A risk of drowning is one of the many hazards which exist at this site. Possible causes listed during the preliminary hazard analysis included falling into the water from a catwalk that has an existing safety rail; falling into the water from a sample collection punt, and; falling into the water while wading out into the stream during sample collection. Listed in order of priority, the highest to lowest would be drowning while 1) wading, 2) punting, and 3) walking on the catwalk where there is a safety rail. Although all three were equal in severity, the probability of occurrence determined the priority. The benefit of each of the three sampling techniques outweighed the risk of drowning given that application of administrative controls (e.g. training/SOP for boat use), PPE (e.g., personal flotation device and/or safety line) and engineering controls (e.g., safety rail present on catwalk) significantly reduced the probability of occurrence to the point where the risk was acceptable considering the benefit. That is assuming conditions do not change from the initial risk assessment. When the sampling actually occurred, a heavy rain had swollen the lake requiring increased outfall flow rate. The individual responsible for sampling the outfall reconsidered the risk of wading given this change in environmental conditions and decided that particular sample would have to wait until the flow subsided. A call to the supervisor confirmed this as the correct course of action. The benefit of collecting the sample after conditions had changed would not justify the risk involved.

3.3.3.1.4 Hazard Recognition, Assessment and Control. The following information is intended to assist in the risk management process by identifying some of the diverse hazards which may be encountered by environmental compliance sampling personnel. Once the inherent hazards of a sampling event are recognized, **Appendix D**, the NAVOSH Reference List, provides a summary, by hazard topic, of current guidance. These references should be considered during the assessment and control phases of the risk management process.

Safety Hazards. Safety hazards are site conditions or possible events that could cause injury to sampling personnel or damage to their equipment. A preliminary site survey and evaluation should be performed to identify safety hazards before the FSP is prepared. The preliminary site survey includes the evaluation of site historical data and identification of all suspected conditions which may pose inhalation or skin absorption hazards or other conditions that may cause death or serious harm. Examples of such hazards include, but are not limited to:

- Confined space entry
- Potentially explosive or flammable situations
- Visible vapor clouds
- Areas where biological indicators, such as dead animals or vegetation, are located
- Pressurized pipes and containers
- Possible electrical shock
- Soil cave-in, excavations and trenching
- Hazards resulting from exposure to chemicals in the media being tested or to chemicals used in performing field tests
- Utilities
- Scaffolding

Chemical Hazards. Chemical hazards present in environmental samples or in the environment being sampled are not the only chemical hazards of concern. Toxic chemicals may also be brought onto a site (e.g., fuels and lubricants for equipment, preservatives for samples, etc.). Hazardous chemicals can be absorbed into the body through various pathways. These pathways include:

- Inhalation of vapors, gases, or particulate (This pathway is typically of the greatest concern for potentially acute exposures.)
- Ingestion of contaminated particulate from hand to mouth contact
- Dermal and eye absorption from direct, unprotected contact, or from exposure to airborne concentrations

Physical Hazards. Potential physical hazards may include, but are not limited to:

- Confined space entry
- Heat and cold stress
- Hazardous flora and fauna (poison ivy, poisonous snakes, stinging insects, animals, etc.)
- Hazardous noise in the area, including that due to use of sampling equipment
- Utilities (gas, electric, water)
- Pressurized lines or containers
- Potential for electrical shock
- Soil cave-in, excavations and trenching
- Powered equipment
- Heavy Equipment
- Explosion and fire
- Radiation Hazards

Task Specific Hazards. Listed below are summaries for some but not all of the known or assumed hazards associated with certain tasks common to environmental compliance sampling. The list of common tasks and associated hazards is not all inclusive but will be expanded with each revision of this manual and as new environmental compliance sampling techniques are developed.

Sediment/Surface Water/Biota Sampling

Chemical
- ° Potential for contaminated material to be splashed onto body or in eyes
- ° Ingestion of contaminated material from hand-to-mouth contact
- ° Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants within the sediments or surface water
- ° Absorption of constituents through the skin

Physical/Environmental
- ° Muscle strain from boring with hand auger
- ° Sampling operations that occur from boats/while wading
- ° Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- ° Skin irritation from contact with insects and vegetation
- ° Interaction with wild animal life

Land Surveying

Chemical
- ° Skin contact with potentially contaminated soil
- ° Ingestion of contaminated material from hand-to-mouth contact

Physical/Environmental
- ° Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- ° Skin irritation from contact with insects and vegetation
- ° Interaction with wild animal life
- °

Geophysical Investigation

Chemical
- ° Skin contact with potentially contaminated soil
- ° Ingestion of contaminated material from hand-to-mouth contact

Physical/Environmental
- ° Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- ° Skin irritation from contact with insects and vegetation
- ° Interaction with native and feral animal life

Surface Soil Sampling/Drive Point Installation

Chemical
- ° Skin contact with potentially contaminated soil or groundwater
- ° Ingestion of contaminated materials from hand-to-mouth contact

Physical/Environmental
- ° Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- ° Skin irritation from contact with insects and vegetation
- ° Interaction wild animal life
- ° Muscle strain from boring with hand auger or hammering of drive point

Monitoring Well Installation

Chemical
- ° Potential for contaminated mud, soil, or groundwater to be splashed onto body or into eyes
- ° Ingestion of contaminated materials from hand-to-mouth contact
- ° Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants
- ° Absorption of constituents through the skin

Physical/Environmental
- ° Heavy objects landing on foot/toe or head
- ° Elevated noise levels from heavy equipment operation
- ° Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- ° Skin irritation from contact with insects and vegetation
- ° Overhead hazards from drill rig operations
- ° Interaction with wild animal life
- ° Contact with underground utility lines

- Muscle strain from lifting hazards
- Explosion from contacting explosive/ignitable materials

Monitoring Well Development

Chemical

- Potential for groundwater, to be splashed onto body or in eyes
- Ingestion of contaminated materials from hand-to-mouth contact
- Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants

Physical/Environmental

- Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- Skin irritation from contact with insects and vegetation
- Interaction with native and feral animal life

Groundwater Sampling/Slug Test/Impact Sampling

Chemical

- Potential for contaminated groundwater to be splashed onto body or in eyes
- Ingestion of contaminated materials from hand-to-mouth contact
- Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants emitting from the well opening

Physical/Environmental

- Skin irritation from contact with insects and vegetation
- Muscle strain from lifting bailer or removing slug
- Cuts from using knives to cut bailer rope
- Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- Interaction with native and feral animal life
- Contact with underground utility lines
- Overhead hazards from drill rig operations

Soil Gas Survey

Chemical

- Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants
- Ingestion of contaminated materials from hand-to-mouth contact
- Skin contact with potentially contaminated material

Physical/Environmental

- Slips, trips, and falls - sloped, uneven terrain, crawling over and under obstacles
- Skin irritation from contact with insects and vegetation
- Contact with underground utilities, fuel lines, etc.
- Interaction with wild animals

Subsurface Sampling - Soil Boring

Chemical

- Potential for contaminated mud, soil, or groundwater to be splashed onto body or in eyes
- Skin contact with potentially contaminated soil
- Ingestion of contaminated soils from hand-to-mouth contact
- Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants

Physical/Environmental

- Elevated noise levels from heavy equipment operations
- Muscle strain from lifting hazards
- Skin irritation from contact with insects and vegetation
- Contact with underground utilities
- Interaction with wild animals
- Heavy objects landing on foot/toe or head
- Explosion from contacting explosive/ignitable materials
- Slips, trips, and falls from sloped, uneven terrain, crawling over and under obstacles

Test Pit/Trenching

Chemical

- Skin contact with potentially contaminated soil
- Ingestion of contaminated materials from hand-to-mouth contact
- Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants
- Skin contact with potentially toxic "pure product" contaminants

Physical/Environmental

- Overhead hazards from backhoe operations
- Skin irritation from contact with insects and vegetation
- Contact with underground utilities
- High grade slopes that may require shoring according to OSHA Standards
- Interaction with wild animal life
- Explosion from contact with explosive/ignitable materials
- Elevated noise levels from heavy equipment operation
- Slips, trips, and falls from sloped and uneven excavation materials or landscape

Drum Sampling

Chemical

- Ingestion of contaminated materials from hand-to-mouth contact
- Inhalation of volatile contaminants or volatile fraction of semi-volatile contaminants
- PPE incompatible with the drum contents
- Skin contact with drum contents

Physical/Environmental

- Mislabeled, multi-labeled, or unlabeled containers that may contain highly contaminated groundwater or soil
- Cuts or abrasions while opening drum or using sampling equipment
- Skin irritation from contact with insects and vegetation
- Interaction with native and feral animal life
- Explosion from introduction of ignition source

Tanker/Roll-Off Box Sampling

Chemical

- Skin contact with potentially contaminated soil or water
- Ingestion of contaminated material from hand-to-mouth contact
- Inhalation of potentially volatile contaminants or volatile fraction of semi-volatile contaminants

Physical/Environmental

- Slips, trips, and falls from elevated heights (i.e., top of roll-off box or tanker) onto ground
- Falling into potentially contaminated material in roll-off box

Stack/Vent/Duct Sampling

Chemical

- Inhalation of fumes, vapors, and dusts that may be toxic or irritating
- Skin and eye irritation from insects, vegetation, fumes, vapors and particulate
- Ingestion of material being sampled and/or sampling reagents from hand to mouth
- Biological exposures to disease carrying animals and their excrements

Physical/Environmental

- Muscle strains from climbing/bending /walking while getting to the sampling site and/or transporting sampling equipment
- Falls, trips, entanglements, and slips from ladders, cages, industrial equipment, masonry obstructions, and safety equipment
- Skin and eye irritation from dusts, fumes, and vapors
- Interactions with wildlife and vegetation
- Electrical shock from power lines, fixed industrial equipment, portable power sources, and lightning
- Suffocation from entry into confined spaces or from breathing the stack plume
- Heat and cold stress from weather extremes, as well as wind

WARNING

When explosive hazards are known to be present, expert advice must be obtained. Consideration must also be given to materials which were originally non-energetic but which may have deteriorated or changed with age to yield an explosive hazard (e.g., peroxides in ethers). Materials may also change sensitivity when dispersed.

3.3.3.2 Health and Safety Elements. The Navy Occupational Safety and Health (NAVOSH) Program, as established by OPNAVINST 5100.23D, protects the health and safety of Navy and DOD environmental compliance sampling personnel. However, the NAVOSH Program must not be considered all inclusive since the hazards and environments encountered during compliance sampling are so diverse. A partnership must exist between compliance sampling personnel and cognizant industrial hygiene and safety professionals to ensure site specific safety and health hazards are identified and addressed.

3.3.3.2.1 HASP Elements. A site specific Health and Safety Plan (HASP), meeting the requirements of **Appendix C**, will be included when the scope of work requires it. See 29 CFR 1910.120(a)(1)(ii) or (iii) to determine applicability. The following is presented as an outline of what you should expect as content in a comprehensive HASP. Not all of the following elements may be required for all compliance sampling events. Each plan must be adapted to be site specific and vary with the extent of hazards anticipated and the operations performed.

I. TITLE PAGE

- Title of document (Health and Safety Plan)
- Sampling program name
- Job number
- Date
- Revision number
- Name, organization, and phone number of Program Manager

II. TABLE OF CONTENTS

III. INTRODUCTION

- Scope and Applicability of the Health and Safety Plan
- Site Entry Procedures
- Procedures for Modifying Health and Safety Plan

IV. KEY PERSONNEL/IDENTIFICATION OF HEALTH AND SAFETY PERSONNEL

- Key Personnel
- Site Specific Health and Safety Personnel
- Organizational Responsibility

V. TASK/OPERATION SAFETY AND HEALTH RISK ANALYSIS

- Historical Overview of Site
  - ° Site Description
    - – Location Description
    - – Land Use
    - – Topography
    - – Climate
    - – Hydrogeology
  - ° Site History
  - ° Current Conditions
- Chemicals Present on Site
- Task by Task Risk Analysis
  - ° Chemical Hazards
  - ° Physical Hazards

VI. PERSONNEL TRAINING REQUIREMENTS

- Pre-assignment Training
- Annual Refresher Training
- Site Supervisors Training
- Training and Briefing Topics
- Heat Stress Training, Recognition, and First Aid
- Cold Weather Training, Recognition, and First Aid

VII. PERSONAL PROTECTIVE EQUIPMENT (PPE) TO BE USED.

- Levels of Protection (i.e. Level A through D)
- Reassessments of Protection Program
- Work Mission Duration
- SOPs for respirator usage

VIII. MEDICAL SURVEILLANCE REQUIREMENTS

- Baseline or Pre-assignment Monitoring
- Periodic Monitoring
- Exposure/Injury/Medical Support

IX. FREQUENCY AND TYPES OF PERSONAL AIR MONITORING/SAMPLING

- Direct-Reading Monitoring Instruments
- Personal Sampling
- Specific Contaminants to be Monitored
  - ° Personnel Air Monitoring Program
  - ° Ambient Air Sampling

X. SITE CONTROL MEASURES

- Site Security
- Buddy System
- Site Communications Plan
- Work Zone Definition
- Nearest Medical Assistance
- Safe Work Practices
- Emergency Alarm Procedures

XI. DECONTAMINATION PLAN

- Personnel Decontamination Procedures
- Levels of PPE Required for Decontamination Personnel
- Equipment Decontamination
- Disposition of Decontamination Wastes

XII. EMERGENCY RESPONSE/CONTINGENCY PLAN

- Pre-emergency Planning
- Personnel Roles and Lines of Authority
- Emergency Recognition/Prevention
- Evacuation Routes/Procedures
- Emergency Telephone Numbers
- Emergency Medical Treatment Procedures
- Fire or Explosion
- Spill or Leaks
- Emergency Equipment/Facilities

XIII. CONFINED SPACE ENTRY PROCEDURES

- Definitions
- General Provisions
- Procedure for Confined Space Entry
- Duties of Confined Space Observer

XIV. SPILL CONTAINMENT PROGRAM

XV. AUTHORIZED CHANGES TO HEALTH AND SAFETY PLAN (section for inclusion of approved modifications)

XVI. Appendices

- Appendix A - Chemicals of Concern: Chemical, Physical, Toxicological, and First Aid Data (MSDS).
- Appendix B - Real Air Monitoring Equipment - Operation, Calibration, and Maintenance Procedures.
- Appendix C - Limitation of Specified Personal Protective Equipment (chemical degradation, permeation, temperature limits).

XVII. LIST OF FIGURES

- Organization chart
- Facility map showing sampling locations
- Map depicting exclusion zones during sampling
- Map of route to nearest emergency medical facility
- Level A decontamination procedure
- Level B and C decontamination procedure
- Level D decontamination procedure
- Equipment decontamination sequence
- Evacuation routes and safe distances
- Facility map with emergency equipment located

XVIII. LIST OF TABLES

- Chemicals of concern (include action levels for Respiratory protection [Permissible Exposure Limit (PEL), Immediately Dangerous to Life and Health (IDLH)], odor thresholds, detection limits of field monitoring equipment)
- Sample PPE inspection checklist
- Levels of PPE planned for sampling tasks
- Available direct reading instruments for air hazard monitoring
- Personnel requirements
- General work practices
- Standing orders for exclusion zone and contamination reduction zone
- Emergency recognition/control measures
- Emergency telephone numbers

NOTE:

> **Appendix C** - *Health and Safety Plan (HASP) Review* provides a thorough HASP review checklist for situations when a site specific "Health and Safety Plan", as required by 29 CFR 1910.120, is necessary. Check with the Environmental Programs Directorate at the Navy Environmental Health Center (COMM 804-363-5500; DSN 864-5500) to make certain the version in this manual is the most current version available.

3.3.3.2.2 <u>Safety Plan Elements</u>. A less extensive and less detailed site specific safety plan will be included for the usual compliance type sampling which does not fall under 29 CFR 1910.120. The vast majority of compliance sampling events will be in this category. This less extensive safety plan, which does not need to meet all the criteria of 29 CFR 1910.120, must still reflect the five step risk management process detailed in **Section 3.3.3.1.2**: Identifying Hazards, Assessing Hazards, Making Risk Decisions, Implementing Controls, and Supervising. The following is presented as an outline of what you should expect a typical safety plan to include. Not all of the following elements may be required for all compliance sampling events. Each plan must be adapted to be site specific and vary with the extent of hazards anticipated and the operations performed.

I. Preliminary Hazard Analysis

II. Site Visit

III. Safety Plan

- Describe (be specific) expected hazards
- List applicable safe operating procedures
- List required (be specific) Personal Protective Equipment (PPE) and any special decontamination and/or disposal requirements
- Conduct (and document) training required to collect samples in a safe manner
- List the warning indications and health effects of each significant toxic contaminant which may be present
- Include a Material Safety Data Sheet (MSDS) for each chemical which would be encountered
- List Points of Contact for cognizant health and safety professionals and supervision for the sampling event

NOTE:

> **Appendix D** - *NAVOSH Reference List* provides pertinent hazard specific information resources, arranged by hazard topic, which compliance sampling personnel and cognizant health and safety professionals should be aware of during the risk management process. Check with the Industrial Hygiene Directorate at the Navy Environmental Health Center (COMM 804-363-5500; DSN 864-5500) if you have questions concerning the references cited or to determine if the cited reference has been updated since publication of this manual.

3.4 <u>EXPLOSIVES SAMPLING</u>.

WARNING

When any sampling operation is to be undertaken which may involve energetic materials, the involvement of explosive trained and certified personnel is mandatory. Explosive safety requirements include use of approved explosive procedures and explosive certified personnel. Section 12.3.3 and other sections of this manual provide amplifying information.

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING AND FIELD TESTING PROCEDURES MANUAL

CHAPTER 4

COMMON SAMPLING PROCEDURES

4.1 PURPOSE. This chapter presents items common to the sampling events covered in **Chapters 5 - 13**. This chapter should be referred to in conjunction with specific sampling procedures discussed in **Chapters 5 -13**. **Section 1.5** and **Figure 1-1** of this manual present a discussion of the relationships among the chapters. Additional references include facility Standard Operating Procedures (SOPs) and special requirements contained in regulatory programs and site permits.

4.2 PREPARATIONS FOR FIELD SAMPLING. The success of a field sampling program depends on the preparation prior to entering the field. Implementation of the Sampling and Analysis Plan (SAP) begins with preparing for the field sampling operation. The following preparation steps should be considered important to the success of the project:

- **Preliminary Off-Site Evaluation.** Prior to implementing the Field Sampling Plan (FSP), the Program Manager and Health and Safety Supervisor should review any Historical Overview and Site Description sections of the Sampling and Analysis Plan (SAP). This review may result in the decision for an on-site evaluation to assess the sampling procedures, relevant safety equipment and personal protective equipment.
- **Equipment Verification.** The FSP should specify an equipment list, including sampling equipment, sample containers, and Personal Protective Equipment (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, conductivity, disinfection chemicals, and temperature require additional field time. The need for additional personnel is based on time demand, 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 is required for most water samples. 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 field work 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 that the samples are analyzed within the required holding times. Holding times are dictated by the regulatory program and data may be invalidated when holding times are not met.

4.2.1 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 U.S. Geological Survey quadrangle map of the study area is essential for sampling in rural areas. Such maps show seldom used trails that may be helpful in gaining access to sampling locations, and indicate the vegetated areas and terrain within the sampling area.

- **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 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.

  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; water, plankton, benthos, fish, vascular plants, wastewater, soil, or solid waste. When the samples are to be collected including nocturnal or daytime sampling. Where within the environmental medium the samples are to be collected, including both horizontal and vertical collections, and 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 knows approximately the types of samples, (liquid, semi-solid, solid, or biological) and the analyses requested on each sample type. Arrangements must be made for the appropriate number of sample containers and preservatives where required. Transportation of samples from the point of collection to the laboratory must be considered, and the Chain-of-Custody Record must be traceable, as detailed in the Quality Assurance Plan (QAP).

- **Equipment.** Prior to going to the sampling location, the sampling gear needs to be checked to ensure that it is correct for the task and in good working order. Label, mark, and otherwise identify all equipment, instruments, reference materials, and associated supplies for measurement processes to indicate calibration or standardization status. Verify all equipment is in working order and that preventative maintenance has been completed according to the SOPs. Expiration dates of reagents and solutions should be checked and verified as to useability.

  If waders are used for surface water sampling, they must be available for the sample collectors. 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 checked out prior to starting the sampling event.

NOTE:

Sampling equipment, when in use, should be anchored to prevent equipment loss in the event the rope or equipment slips through the hands of the sample collector.

- **Safety.** Safety of sampling personnel is paramount. During wading operations, a rope should be attached to the sampler and extend to an anchor person on shore. In boating operations, the sampling boat should not be overloaded, but at least two people are required in the boat, one to collect the samples and one to operate the motor. Boat personnel, by regulation, are required to wear life preservers. When collecting samples, beware of poisonous snakes, nests of wasps or bees, ticks, and other animals that potentially may cause injury to sample collectors. Refer to **Section 3.3.3** and **Appendix D** for specific hazard identification and appropriate reference for detailed control measures.

WARNING

Refer to Section 3.3.3 and Appendix D for specific hazard identification and appropriate reference for detailed control measures.

- **Personnel Transportation and Lodging.** The Program Manager must consider arrangements for transporting sampling personnel and equipment to the sampling site, and for lodging accommodations when the sampling extends beyond a working day.

4.2.2 Preliminary On-Site Evaluation. When sampling for the first time at a new sample 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 modify the expected hazards or FSP.

4.2.3 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, concentrate first on identifying conditions which present an acute health hazard, and then on evaluating exposure to chemicals such as carcinogens that could create long term health problems.

The first priority, if samples are to be collected in a confined spot, shall be testing the air within the space for oxygen content. Next, tests for explosive levels of flammable vapors shall be conducted, 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.

Second, 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.

4.2.4 Explosive Safety Evaluation.

WARNING

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

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

4.2.5 Preliminary Sampling Evaluation. Sampling locations should be inspected to ensure the information in the FSP is correct. All equipment should be checked the day before the sampling event to ensure all maintenance, parts, and records are available for the sampling operation. Preventative maintenance should be performed, if needed.

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

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.

4.3 THE SAMPLING EVENT. A sampling event would 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 sampling containers.
- Set-up equipment, work areas and safety areas, as described in the FSP.
- Collect samples at the locations specified in FSP or reference procedure.
- Immediately following sample collection, ensure that each sample container is labeled as described in the FSP. The sample label must be traceable to the sample number, date, time sampled, sampler's initials, preservative, and site name/location or unique project identifier.
- Document the exact location of the collected sample(s) in the **Field Log Book** or **Field Notes**. Also, record in the **Field Log Book** or **Field Notes** 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 be recorded.
- Preserve and/or ice samples as appropriate and record preservation method.
- Perform field tests or field screening measurements and record all observations.
- Complete the Chain-of-Custody Record and other field records.
- Pack and seal the shipping container with collected samples, and transport the shipping container with the Chain-of-Custody Record and any laboratory required forms to the laboratory. Retain copies of transmitted forms.
- Return all forms and copies of the relevant Field Log Book/Field Note pages to the Program Manager or designee.
- Clean sampling equipment for the next sampling event or storage until next event.
- Breakdown all work area and safety areas as required and return the site to condition found at the start of the sampling event.
- Dispose of all waste materials using appropriate procedures.

4.4 SAMPLING PROCEDURES. The FSP 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 (the collection of initial runoff samples after a rain for contamination)
- Sample handling procedures for each sample container (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 properly used materials
- Equipment decontamination procedures to be used between sample locations and between sampling events
- Record keeping requirements, documentation handling and retention requirements

- Sample, equipment and materials storage requirements
- Provisions for storage and/or disposal of wastes generated during field sampling

4.4.1 Sampling Strategies. See **Appendix E** for sampler and sampling recommendations and strategies for waste materials. For sampling during clean-up or site investigations see the Navy Installation Restoration (IR) program. Sampling strategies for drinking water, wastewater, groundwater and Toxic Substances Control Act (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.

4.4.2 Sampling Procedure Checklist. 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, start-up, shut-down
  - [ ] Maintenance
  - [ ] Climate
  - [ ] Hazards
- **Equipment**
  - [ ] Maintenance
  - [ ] Preparation and Cleaning
  - [ ] Operation
  - [ ] Calibration/Standardization
- **Sample Handling, Transportation, Storage and Preservation**
  - [ ] Chain-of-Custody
  - [ ] Seals
  - [ ] Forms
  - [ ] Containers
  - [ ] Preservatives, Reagents, and supplies
- **QA/QC**
  - [ ] Controls on process
  - [ ] Audits
  - [ ] Training
  - [ ] Samples, Blanks, Duplicates, 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 Quality Control
  - [ ] Reporting format and schedule

4.5 SAMPLE DOCUMENTATION AND CHAIN-OF-CUSTODY PROCEDURES. Thorough documentation is required to support sample validity. The documentation must verify that the samples are representative, were collected in accordance with the requirements of the FSP, and are not vulnerable to tampering before being received by the laboratory.

Sample documentation and Chain-of-Custody procedures include:

- A completed sample collection label attached to all sample containers.
- Records of sampling operations written in Field Log Books, Field Notes or related forms as designated for the operation in the SAP. Records include sample type, sample matrix, sampling method, field test methods and quality control procedures. A table may be used to present this information.
- Identification of every sample container on a Chain-of-Custody 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.

4.5.1 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.

The **Program Manager** shall establish a system for assigning a unique sample number to each sample collected in the field. The numbering system shall be described in the FSP (in case additional samples are generated in the field). The number for each sample shall be used to identify the sample on the **Field Log Books**, **Field Notes**, sample container, and on the Chain-of-Custody Record. The number may be used on other forms and reports presenting measurements, test data or evaluations.

The sample number provides a common identifying code of 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
- Chain-of-Custody Record number
- Matrix
- Location
- Sample type
- Sample date
- Sample time
- Sampler name
- Parameter
- Analytical result
- Quality control data
- Compliance limit
- Data qualifier code (Optional)

Results from analysis of Trip Blanks, Field Blanks, Equipment Decontamination Blanks, Split Samples and Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples may be entered into a computer data base. 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 sample number consist of elements describing the sample type, matrix, location, and the time and date of sample collection as required to assign a unique number to each sample. For example, a sample number may consist of one or all of the following elements:

"S-T-MM-Q-L-D"

where:

S = Site Code (facility)
T = Type
  S = grab
  X = composite
  C = continuous
MM = matrix
  SO = soil
  SL = sludge
  SS = solid waste
  GW = groundwater
  SW = surface water
  DW = drinking water
  OW = discharge water
  D1 = drum liquid organic (floating liquid)
  D2 = drum liquid water (second liquid phase)
  DS = drum sludge or bottom solids
  AA = ambient air
  ST = stack gas
Q = Quality Control Code:
  P = Test or Duplicate
  B = Field Blank or Equipment Decontamination Blank
L = Location Code
  Description code shown on site map:
    XX/YY/ZZ = Site Grid Coordinates
      North in feet (two digits) /
      East in feet (two digits) /
      Depth below surface in feet (two digits)
    DR### = Drum Number
      Alpha numeric designation corresponding to a sampling location indicated on the site
D = Date or Sequence Number of Sample

NOTE:

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

4.5.2 Sample Container Labeling. Sample labels are an important part of proper documentation to reduce the possibility of confusing sample containers, and provide the information necessary during handling. 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. Sample labels should include sample number, date/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, lot controlled and traceability from container preparation, preservation chemicals, sampling and testing is required.

The labeling of sample containers is done by a designated Field Sample Custodian or sampler when the containers are filled. Preprinted adhesive, multiple part labels formatted as shown in **Figure 4-1** may be used. Each part includes the unique sample container number that may be prenumbered to avoid duplication.

When a sample container is filled in the field, one part of the label is applied to the container, a second part is signed and applied to the Custody Form, and the last part placed in the Field Log Book/Field Notes by the person responsible for collecting the sample(s).

NOTE:

Label entries should be made using waterproof ink. Field Log Book/Field Notes notations should explain if a pencil was used to fill out the Sample Container Label because the ballpoint pen would not function due to field weather conditions.

PROJECT NAME

Custody or Tracking #: XXX

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

Figure 4-1 Multiple Part Container Label

4.5.3 <u>Field Log Book/Field Notes</u>. The **Field Log Book/Field Notes** is the written record of all field data, observations, field equipment calibrations, and sample collection activities. Potential for future legal actions dictates that the **Field Log Book/Field Notes** be site-specific, and that they be bound (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 in using Field Log Books/Notes:

- The **Field Log Book/Field Notes** shall be assigned to the **Quality Assurance/Quality Control (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 Log Book the individual to whom it was assigned. The log books may be controlled by the QA/QC Coordinator or the Program Manager.
- Each Log Book shall be annotated with the sampling program name or number.
- List key personnel and telephone numbers on the first page.
- Entries shall be written in waterproof blue or black ballpoint pen. Avoid felt tip pens.
- 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 and/or facility. Include sketches of layout, structural features, and points of interest or contamination. Include north arrow and rough scale. If possible, obtain a site map (reduced if necessary) and permanently place it in the Log Book/Notes.
- 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.
- Do not erase or scratch out. Mistakes shall be lined out with a single line through the error, corrected material inserted, initialed and dated by the person who made the error and the reason for the error annotated.
- Entries or corrections made by individuals other than the person to whom the Log Book was assigned shall be dated and signed by the individual making the entry or correction. An explanation for the correction should be annotated.
- The last entry for each day should include a short summary of the day's activities, weather conditions and the time you leave the site. 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 Log Book/Notes 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 Field Log Books/Field Notes shall be returned to the individual designated for review and final storage when sampling is completed as described in the QAP.
- Log Book/Notes entries will contain a variety of information. Information which should be entered at the start of each day of sampling includes:
    - Date(s) of the sampling event
    - Time sampling started and approximate time for set-up of equipment

  - Weather
  - Level of personal protective equipment (PPE) being used
  - Names of field sampling team members and others present during the sampling
- Fully document all deviations from the FSP 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 Field Log Book/Field Notes.
- 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 repeatability of future sampling events. Measurements from permanent features (center line of road, numbered utility pole, etc.) to the sample point must be made and entered into the Field Log Book/Field Notes. 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 and roll number. The record is logged in the Field Log Book/Field Notes to identify which sampling site is depicted in the photograph.

NOTE:

The film roll can be identified by taking the first photograph of a informational sign with the sampling program name, number, and the film roll 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 with all information recorded shall be put into the Field Log Book/Field Notes.
- 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 Field Log Book/Field Notes.
- Decontamination and/or disposal procedures for all equipment, samples, protective clothing, and personnel decontamination procedures should be noted.
- For each delivery or shipment of samples to a laboratory, record the following information in the Field Log Book/Field Notes:
  - 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

4.5.4 <u>Field Notes</u>. Field Notes or Field Sampling Forms are used in addition to or in lieu of a Field Log Notes. When Field Notes are used in lieu of a Field Log Book, the record keeping practices presented in **Section 4.5.3** 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 and assist the sampler 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 4-2**.

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 bound logbooks.

Field Form
(Example for Well Monitoring Data)

WELL REPORT FORM

Company/Command:__________________________ Code: _________ Project #: ____________________

Site Name:______________________________ Date Sampled:__________ Sampled by: __________

****

Well ID ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Well Depth (ft) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Water Level (ft) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Water Ht. (ft) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Vol Purged (gal) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Date/time ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Analyst ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

pH (units) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Conductivity
(Umhos/cm) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

Temp (C) ______ ______ ______ ______ ______ ______ ______ ______ ______ ______

****

Filtered Y/N Y/N Y/N Y/N Y/N Y/N Y/N Y/N Y/N Y/N

in the (Field/Lab) through (#40 Whatman / 0.45 micron/Other _____________)

Samples were collected with: [ ]shallow well [ ]submersible [ ]bailer
[ ]peristaltic [ ]___________ [ ]______
Depth Measurement Method ______________________

Weather Conditions:____

Number/Type bottles collected: ______1/2 gal plastic ______liter plastic
______liter glass ______GC vials

Notes or Problems encountered:

Figure 4-2 Field Form

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 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 FSP. 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 and 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.

4.5.5 **Chain-of-Custody.** 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 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 Chain-of-Custody Record that is completed by the Sample Custodian.

4.5.5.1 **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 chain of custody is broken.

The Program Manager or designee shall review all field activities to determine whether proper custody procedures were followed during the field work and whether additional samples are required. The Sampler or Sample Custodian shall notify the Program Manager of any breach or irregularity of chain-of-custody procedures described in the QAP or FSP.

4.5.5.2 **Chain-of-Custody Records.** There are many transfers of custody during the course of a sampling program, from time of collection through final sample disposal. All sample containers must be accompanied by a Chain-of-Custody Record to document these transfers. A separate Chain-of-Custody Record shall be prepared by the Field Sample Custodian or sampler for each sampling event. In some programs a Chain-of- Custody Record accompanies each shipping container and includes a prenumbered Chain-of-Custody 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.

Two examples used for a sample Chain-of-Custody Record are shown in **Figure 4-3**. The examples present actual documents being used within commands to trace sample custody and meet project specific needs.

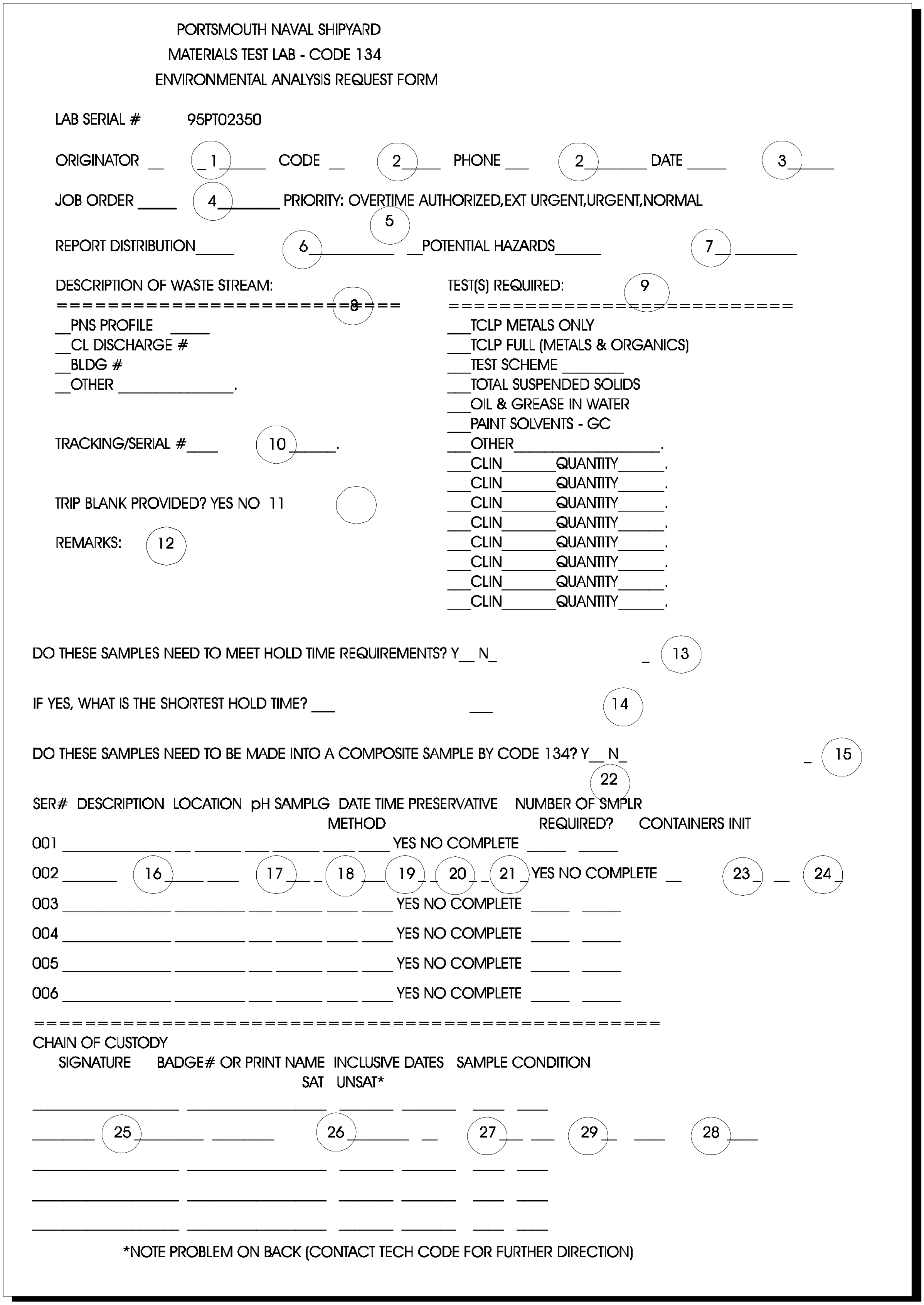
Example 1 of **Figure 4-3** is a project specific form used for Resource Conservation and Recovery Act (RCRA) characterizations of known waste materials. The form is serially numbered to ensure unique identification. The following information relates to the numbered blocks:

Chain of Custody (COC) - Example 1.

Section 1 to 7 are completed by The Program Manager, designee or sampler:

(1) **Originator** is the Program Manager or Designee from the SAP.

(2) The **Code** or Command name and **Phone** number of the Designee



PORTSMOUTH NAVAL SHIPYARD
MATERIALS TEST LAB - CODE 134
ENVIRONMENTAL ANALYSIS REQUEST FORM

LAB SERIAL # 95PT02350

ORIGINATOR __ ① ____ CODE __ ② ____ PHONE ___ ② ______ DATE _____ ③ _____

JOB ORDER _____ ④ ______ PRIORITY: OVERTIME AUTHORIZED,EXT URGENT,URGENT,NORMAL ⑤

REPORT DISTRIBUTION_____ ⑥ __________ __POTENTIAL HAZARDS______ ⑦ __ ________

DESCRIPTION OF WASTE STREAM: ⑧
==========================
__PNS PROFILE _____
__CL DISCHARGE #
__BLDG #
__OTHER ______________.

TRACKING/SERIAL #____ ⑩ _____.

TRIP BLANK PROVIDED? YES NO 11

REMARKS: ⑫

TEST(S) REQUIRED: ⑨
==========================
___TCLP METALS ONLY
___TCLP FULL (METALS & ORGANICS)
___TEST SCHEME ________
___TOTAL SUSPENDED SOLIDS
___OIL & GREASE IN WATER
___PAINT SOLVENTS - GC
___OTHER_________________.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.
___CLIN_______QUANTITY______.

DO THESE SAMPLES NEED TO MEET HOLD TIME REQUIREMENTS? Y__ N_ _ ⑬

IF YES, WHAT IS THE SHORTEST HOLD TIME? ___ ___ ⑭

DO THESE SAMPLES NEED TO BE MADE INTO A COMPOSITE SAMPLE BY CODE 134? Y__ N_ _ ⑮

㉒

SER# DESCRIPTION LOCATION pH SAMPLG METHOD DATE TIME PRESERVATIVE REQUIRED? NUMBER OF CONTAINERS SMPLR INIT

001 _____________ __ ______ ___ ______ ____ ____ YES NO COMPLETE _____ _____
002 ______ ⑯ ____ ____ ⑰ __ _ ⑱ __ ⑲ _ _ ⑳ _ _ ㉑ _ YES NO COMPLETE __ ㉓ _ __ ㉔ _
003 _____________ ________ ___ ______ ____ ____ YES NO COMPLETE _____ _____
004 _____________ ________ ___ ______ ____ ____ YES NO COMPLETE _____ _____
005 _____________ ________ ___ ______ ____ ____ YES NO COMPLETE _____ _____
006 _____________ ________ ___ ______ ____ ____ YES NO COMPLETE _____ _____

===

CHAIN OF CUSTODY
SIGNATURE BADGE# OR PRINT NAME INCLUSIVE DATES SAMPLE CONDITION SAT UNSAT*

_________________ ________________ _______ _______ ____ ____
_______ ㉕ ________ _______ ㉖ _______ _ ㉗ __ ___ ㉙ _ ___ ㉘ ___
_________________ ________________ _______ _______ ____ ____
_________________ ________________ _______ _______ ____ ____
_________________ ________________ _______ _______ ____ ____

*NOTE PROBLEM ON BACK (CONTACT TECH CODE FOR FURTHER DIRECTION)

Figure 4-3 Chain of Custody Record (Example 1)

(3) **Date** of the sampling event.

(4) **Job Order** number is the funding number provided by the Program Manager.The **Priority** for sample processing designated by the Program Manager or the holding time requirements of the regulatory program. Four priorities are routinely ordered which authorize the overtime costs necessary for completing the testing. Approval signatures authorizing overtime must be obtained.

(6) **Report Distribution** include the Program Manager and any other personnel designated in the SAP.

(7) **Potential Hazards** are listed by category name such as corrosive, flammable, sanitary sewage, toxic, carcinogenic or unknown.

Sections 8 to First line of 25 to 28 are completed by the sampler:

(8) The **Description** of the material being sampled is recorded as to profile number, discharge number, building number or other identifier used for the project.

(9) The **Test(s) Required** by the SAP are referenced to the SOP number or marked next to the test. Specific test names designated by the laboratory should be reviewed to ensure conformance to the SAP.

(10) Individual waste streams are coded to track the quantities of waste. Enter the **Tracking/Serial #** codes for the waste stream sampled.

(11) The **Trip Blank** is only necessary when analyzing for volatile materials. Indicate when a Trip Blank is provided and the source of the trip blank material.

(12) Space is provided for additional **Remarks** or to further clarify information about the samples. The Quantity and Type of container are often listed in this area when required by the SAP.

(13) This form allows the option for meeting holding times by indicating **Yes** or **No** to the question, **"Do these samples need to meet hold time requirements?".**

(14) Hold time indicates when the holding time is required to be met. The question, **"What is the shortest hold time?**" is indicated to ensure the testing is started in time to meet the holding time requirements.

(15) **Do these samples need to be made into a composite sample by Code 134?** indicates a specific program requirement for compositing the sample in the laboratory.

(16) A Description of the sample includes waste stream identity, type of material or source.

(17) The **Location** of the sample to exactly pinpoint the site such as west side of pile middle is noted.

(18) The **pH** taken in the field is recorded here for each sample. If no pH is taken "**NA**" is written in the column.

(19) The **Sampling Method** is indicated by reference to an SOP number or the equipment used.

(20) The **Date** the sample is collected.

(21) The **Time** the sample is collected.

(22) The **Preservative required** and if completed by the sampler is indicated by circling **Yes** or **No** and **Completed**.

(23) The **Number of Containers** used for the sample is indicated.

(24) Enter the **Sampler's Initials**. If more than one sampler is involved in the collection all should initial where indicated.

(25) The **Signature** of the Sample Custodian or Sampler is entered to initiate documented custody.

(26) Enter the **Badge #** or **Print Name** for readability.

(27) Enter the date the sample was collected on the first line, first column, named **Inclusive Date** which indicates the start of custody.

(28) Note the **Sample Condition** when signed by indicating **SAT** for Satisfactory or **UNSAT** for unsatisfactory. Note on the back of the document any deviations or problems with the sample(s).

Section 29 and the following lines of 25 to 28 are completed when custody of the samples are transferred to other individuals:

(29) The person surrendering custody enters the date the sample was relinquished in the first line second column named **Inclusive Date**. The person receiving custody completes lines 25 to 28 until custody is relinquished. All samples indicated on the form must be accounted for and in satisfactory condition when custody is received. Unsatisfactory or missing samples are noted and the Program Manager is notified promptly, in writing.

Example 2 of **Figure 4-3** is used for a wide variety of regulatory programs and meets legal chain of custody requirements. Example 2 tracks the samples from sample collection to disposal. All sampling, preservative and test information is included. The FSP will indicate the individual responsible for completing each section. The following information relates to the numbered blocks:

<u>**Chain of Custody (COC) - Example 2**</u>

(1) The **Company/Command** name and **Code** for the source of the funding are entered.
(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** is indicated 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 is entered. (See Section 18)
(9) The initials for the person **Sampled By** are entered.
(10) The code for Sample **Matrix** such as liquid, solid, and gas is entered. (See Section 18)
(11) The code for **Preservative** is entered. (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 in listed and may reference descriptions in the QAP.
(14) The field reading for **pH** is entered for the sample containers indicated.
(15) The field reading for **Temperature** with the unit of measure is entered for the sample containers indicated. The FSP 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 FSP. This verification may be temperature, pH or if all is correct an indication is made as "OK". Chemical and physical changes may take place during transport to the laboratory. The QAP may require verification of preservation by the laboratory upon receipt.
(18) This section of the custody form are **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. During sampler training the codes are explained and the interpretation defined.
(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.**) is entered for testing work performed by a designated contract laboratory.
(25) The **Sample Disposal** method is indicated and the date completed is filled in.
(26) The signature and **Company/Command** of the person relinquishing custody (**Relinquished By**) is entered.
(27) The signature of the person custody is received (**Rec'd**) **By** is entered.
(28) The **Date/Time** custody is transferred is indicated.

The Chain-of-Custody Record does identify 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 quality control (QC) samples. Based on the needs and data use, the Chain-of-Custody 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 and wastewater testing does 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.

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 and wastewater testing does 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 Chain-of-Custody Record may be prepared for the second set of samples.The additional set of Chain-of-Custody 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 duly noted.

Upon completion of the packing of each shipping container, the Field Sample Custodian shall confirm the completeness of the Chain-of-Custody Record by signing the Chain-of-Custody 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 Field Log Book/Notes or copy of the Field Form.

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

After the Chain-of-Custody 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.

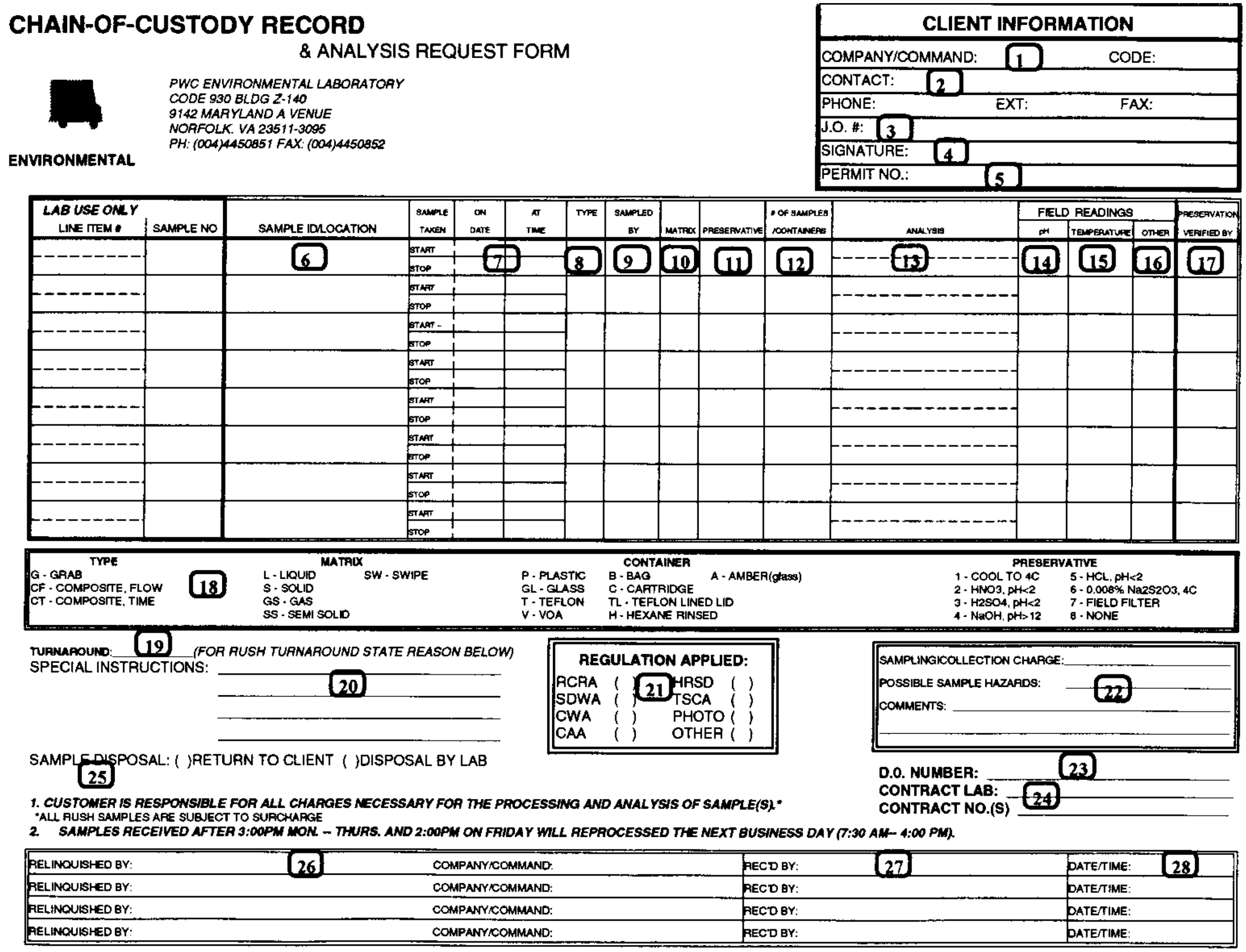
Field chain of custody terminates upon laboratory receipt of the samples. The laboratory should complete the "received by" sections and if appropriate, the "preservative checks" sections on the Chain-of-Custody Record and return the original signed record to the Program Manager. If there are any discrepancies between the Chain-of-Custody Record, the contents of the shipping container, and the QAP 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 record keeping to ensure adherence to the SAP. This added check is often used in drinking water and wastewater testing program for compliance monitoring. Full copies of the SAP may not be disclosed to the laboratory, but record keeping and information checks may still be performed by the laboratory by listing the checks in the contract to ensure the samples received meet the requirements of the SAP.

4.5.5.3 <u>Custody Seals</u> (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 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.

4.5.5.4 <u>Custody Transfer</u>. Transfer of custody and shipment procedures are as follows:

- Each sample shipping container shall be accompanied by a properly completed Chain-of-Custody 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 send a copy of the Chain-of-Custody 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/from a secure storage area.



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: CODE:
CONTACT:
PHONE: EXT: FAX:
J.O. #:
SIGNATURE:
PERMIT NO.:

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	FIELD READINGS TEMPERATURE	FIELD READINGS OTHER	PRESERVATION VERIFIED BY
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												

TYPE
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:______*(FOR RUSH TURNAROUND STATE REASON BELOW)*
SPECIAL INSTRUCTIONS: ____________

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

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

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

D.O. NUMBER: ______
CONTRACT LAB: ______
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:	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:
RELINQUISHED BY:	COMPANY/COMMAND:	REC'D BY:	DATE/TIME:

Figure 4-3 Chain of Custody Record (Example 2)

- 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 Chain-of-Custody Record as long as it is sealed inside the package with the sample container and the custody seals remain intact.

4.5.6 <u>REQUEST FOR ANALYSIS</u>. In more complex sampling programs an additional form may be used to request testing. The Request For Analysis Form is often incorporated into the Chain-of-Custody Record since the Chain must accompany the samples.

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 may 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 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.

4.6 <u>SAMPLE PACKAGING, HANDLING, AND TRANSPORTATION</u>. 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 is regulated by the U.S. Department of Transportation (DOT). Instructions for classification, labeling, and packaging of hazardous materials are contained in DOT regulations (49 CFR 172 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 step-by-step procedure for determining the proper classification and shipping data for samples is presented in **Appendix F**. A summary of specific requirements are addressed below. Should questions arise, assistance is available from the DOT and Federal Aviation Administration hotlines, which are listed in **Appendix G**.

Samples obtained at sites are classified for shipping purposes as either environmental (non-hazardous) 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 your 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 Reportable Quantities (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 Reportable Quantity, *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.

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***) provided that the following requirements are met:

- **Samples for Analysis** – 40 CFR 261.4(d): A sample of solid waste or a sample of water, soil or air, which is collected for the sole purpose of testing to determine its characteristics or composition, when samples are being sent to the laboratory for testing or are being returned to the collector after testing. The details of 40 CFR 261.4(d) are in Appendix G.1.

- **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. The details of 40 CFR 261.4(e) are in Appendix G.2.

4.6.1 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 which 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 4-1** lists sample packaging procedures that will ensure samples arrive at the laboratory with the Chain-of-Custody 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.

4.6.1.1 Samples Classified as Flammable Liquid. Table 4-2 Column 1 lists packaging procedures which 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 N.O.S.** (N.O.S.= Not Otherwise Specified), 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 than 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 three minute period, or a material having one gram or more liquid separation, when determined in accordance with the procedures specified in *ASTM D4359-84, Standard Test Procedure for Determining whether a Material is a Liquid or Solid, (49 CFR 171.8).*

4.6.1.2 Samples Classified as Poison DOT Class 6.1. Table 4-2 Column 2 lists packaging procedures which 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 (which is usually diluted with trichlorobenzene), and many of the other poisonous materials commonly encountered.

4.6.1.3 CERCLA Reportable Quantities - DOT Class 9. Table 4-2 Column 3 lists packaging procedures 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 Substance where the quantity of material in each container *exceeds* the CERCLA Reportable Quantities 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 Chain-of-Custody Record for each group of containers that is being shipped at the same time to the same destination. List the container numbers on the Chain-of-Custody Record.

Table 4-1 Packaging 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 lbs.	66 lbs.	66 lbs.
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 ltrs. (1 gal.) *Solids* - 5 kgs. (11 lbs.)	*Liquids* - 4 ltrs. (1 gal.) *Solids* - 5 kgs. (11 lbs.)
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 mil 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 which will prevent bottle breakage.	3	*Liquids:*	
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	3	
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		
Place the canned or bagged sample containers in the cooler. If plastic bottles are being used, alternate them with any glass container.		5	4
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 **Chain-of-Custody 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 **Chain-of-Custody Record**.	9	8	7
Seal the cooler lid closed with clear tape or strapping tape. Affix security seals.	10	9	8

* Numbers indicate the instructions that must be followed.

Table 4-2 Packaging Not by Common Carrier

Instructions	Not by Common Carrier	
	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 may be read.		3
Place about ½" 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" of cushioning material (e.g., vermiculite or plastic foam) in the bottom of a watertight metal or equivalent strength plastic shipping container.	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 **Chain-of-Custody 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
If the shipping container used is a picnic cooler, tape the drain plug closed so it will not open.	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.

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.

4.6.2 Marking/Labeling. The 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 ppm PCBs. This requirement applies to sample containers and to 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, n.o.s., NA3082
    - solid, n.o.s., NA3077
- If EPA Hazardous Waste Manifest *is not* required:
  - ° Environmentally hazardous substances,
    - liquid, n.o.s., UN3082
    - solid, n.o.s., 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 the **National Fire Protection Association (NFPA)**, the **Hazardous Materials Identification System (HMIS)**, **American National Standard Institute (ANSI)**, and the **Department of Transportation (DOT)**.

4.6.3 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 three 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.

4.7 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROTOCOL. **Quality Control (QC)** is a normal part of good field and laboratory practice. QC includes all of the procedures applied to data collection and generation activities in order 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 (sampling and analytical methods, use of Field Blanks, Field Duplicate samples, inclusion of performance or Standard samples, statistics, 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.

Quality Assurance (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 data quality objectives 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 Quality Assurance/Quality Control (QA/QC) Protocol is specified in the **Quality Assurance Plan** for each job that involves field sampling. QA/QC requirements are based on the level of data reliability required for the project, and may address specific regulatory requirements. The purpose of QA/QC protocol is to ensure that:

- The laboratory receives samples which 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 purpose include training of personnel, providing detailed procedures for preparation, collection, marking/handling, packaging, packing, transfer of samples, and validation and verification of the administrative process and sampling techniques.

4.7.1 Decontamination of Sampling Equipment. The **FSP** 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.

4.7.2 Sample Container Cleanliness Requirements. Sample containers are a possible source of sample contamination. The **Quality Assurance Plan (QAP)** 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 Field Log Book/Notes or 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 Safe Drinking Water Act (SDWA) and Clean Water Act (CWA) parameters may require laboratory cleaned containers proven to be below the limit of detection for the method.

In some clean sample matrices sample containers are reusable. If sample containers are re-used, they should be decontaminated and **Field Blank** samples should be submitted to the laboratory to verify container cleanliness prior to use in the field operation.

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 being made in the Field Log Book/Notes.

4.7.3 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 QAP. General sample container requirements are shown in **Appendix H** for different matrices and analytical parameters. Compliance with specific instructions in the FSP 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.

4.7.4 Sample Preservation and Storage Requirements. Special preservation and storage requirements should be specified in the FSP 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 H**. The specific requirements of the FSP 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 QC sample leaving the laboratory.

4.7.5 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 and/or analysis. Holding time limits summarized in **Appendix H** are method and program requirements. Site specific holding times specified in the FSP take precedence.

4.7.6 Laboratory/Field Analytical Procedures. Laboratory analytical procedures for each parameter are specified based on the compliance limits, permit limits and data needs stated in the Quality Assurance Plan. The QAP or Custody Record indicates to the laboratory which sample containers are to be analyzed for what parameters and specifies the analytical methods. Field testing requires the same level of Quality Control (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 Field Log Book/Notes or Field Form and the Program Manager must be informed immediately of sample numbers affected.

4.7.7 Quality Control (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 are representative of the site or matrix sampled. A number of different QC samples may be specified. Each of the following types check for a potential problem which can affect data reliability. The recommended frequency for each type of QC sample is summarized in **Appendix I**.

4.7.7.1 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 be different sample container numbers or designations to indicate variations made to the samples.

NOTE:

> The term "test sample" is synonymous with the general term "sample" mentioned throughout the text. Test samples are those samples taken from the field for analysis.

4.7.7.2 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 conditions. Field Duplicate samples are:

- assigned different container numbers
- specified in the Field Log Book/Notes or on the Field Form
- distinguished from the test samples on the Chain-of-Custody Record or Field records
- often submitted blind as to designation so the laboratory data assures objectivity

NOTE:

> Exception. Each test sample collected for a specific organic analysis may consist of two containers filled with the same material; these may be given different container numbers but are designated as the same sample on the Chain-of-Custody Record. Only one 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 four containers, with two pairs of containers being designated on the Chain-of-Custody 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 extraction and analytical procedures.

The containers may be submitted to different laboratories for identical analyses to obtain information on inter-laboratory repeatability and field extraction. This is a **Split Sample**.

4.7.7.3 Equipment Decontamination Blanks. Equipment Decontamination Blanks provide information on the levels of cross-contamination resulting from field or laboratory sample preparation actions. These blanks are specified in the **FSP** and on **Field Sampling Forms**, and are prepared in the field. An **Equipment Decontamination Blank** is an organic or aqueous solution 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:

- A **Field Groundwater** Equipment Decontamination Blank, for metals analysis consists of ASTM Type II water or better that is handled by the bailer, filtered, placed in a sample container, and preserved using the same procedures as used for the test and duplicate samples.
- A **Soil Sampling** Equipment Decontamination Blank for semivolatile organics analysis consists of rinsing the field equipment prior to its use and collecting the solvent or materials for analysis.
- A **PCB Wipe Sample** Equipment Decontamination Blank consists of a wipe pad used to wipe the sampling template in the same way that the pad is handled during the actual wipe sampling of a surface.

One Equipment Decontamination Blank is collected for each type of equipment used in sampling 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 **Chain-of-**

Custody Record. More blanks may be collected depending on the data quality needs.

4.7.7.4 Field Blanks. Field Blanks are prepared and analyzed to check cleanliness of sample containers, environmental contamination, purity of reagents or solvents used in the field. A sample container is filled with laboratory ASTM Type I or II water, preserved, and is submitted for analysis for 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.

4.7.7.5 Trip Blanks. A Trip Blank is used with **Volatile Organic Analyte (VOA)** analysis of water. A blank may consist of two **40-mL 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 silicone rubber and teflon septums. 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 FSP 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 Chain-of-Custody Record.

4.7.7.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD). Project or compliance QC procedures require that the laboratory spike a portion of the matrix at a frequency dependent on the heterogeneity of the sample matrix or laboratory certification requirements, with a predetermined quantity of analyte(s) prior to sample extraction/digestion and analysis. The frequency of performing a matrix spike is dependent on the data quality needs.

For a matrix spike duplicate, a second portion of the matrix is spiked. The need for performing a matrix spike duplicate is regulatory dependent.

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

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 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** material on the Chain-of-Custody Record.

The MS/MSD samples are commonly used in CERCLA testing, but are not commonly used in CWA or SDWA testing. Matrix Spikes are routinely performed by the laboratory as part of its internal quality control on randomly chosen samples. If matrix spike data is required for SDWA or CWA reporting requirements, a request must be made to the laboratory to ensure the matrix spike is performed and reported on the appropriate sample.

4.7.8 Field Audits. The QAP 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:

- **FIELD RECORDS**
  - Chain-of-Custody Records
  - Sample Container Labels
  - Log Books 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 security seals

4.8 SAMPLE EQUIPMENT LIST. Equipment specific to each type of media is found at the end of the related chapters. For a complete listing of manufacturers and distributors of sampling equipment, refer to *The Green Pages* published by Delphos International and endorsed by the EPA. For more information, call Delphos International on (202) 337-6300. The following is a generic Sample Equipment List:

- ☐ Review by Health and Safety Supervisor
- ☐ Review by Team Leader
- ☐ Review Arrangements for Sampling with Sampling Members Including Transportation, Safety Procedures and Emergency Operations
- ☐ Review Arrangements for Sample Transportation and Waste Material Disposal
- ☐ Review Arrangements for Testing with Laboratory
- ☐ Equipment and Material Inventory
- ☐ Map of Sampling Location(s)
- ☐ Sampling SOP
- ☐ Field Log Book or Field Form
- ☐ Pen(s)
- ☐ Containers
- ☐ Preservatives
- ☐ Labels
- ☐ Markers
- ☐ Coolers
- ☐ Ice
- ☐ Packing Material
- ☐ Packaging Tape
- ☐ Chain-of-Custody 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
- ☐ Lab Instructions (if different from Custody Form)

CHAPTER 5

SOIL SAMPLING

5.1 PURPOSE. This chapter identifies the major steps and technical areas that will be required/ encountered in conducting a soil sampling program. The role of the sampling run is defined along with procedures that will guide the sampler in conducting a compliant program. The soil sampling program presented does not address site clean-up or investigation. For related information, refer to the Navy Installation Restoration (IR) Program.

5.2 SCOPE. The characterization of soil contamination requires that samples be collected from identifiable locations and that samples received by the laboratory represent actual soil conditions. Soil sampling programs:

- Determine site **lithology** (presence and location of different types of soil, bedrock, or groundwater).
- Define the extent, depth, type, and severity of soil contamination.
- Determine soil "cleanliness" during underground storage tank (UST) removal, polychlorinated biphenyl (PCB) transformer operation, or background monitoring at a treatment, storage, and disposal facility (TSD) or hazardous waste handling areas.

Sampling personnel are critically important to sampling program success, since the sampler is often in the best position to detect areas of suspicion. Even though sampling techniques may be sophisticated, these should not be relied upon to replace good judgment and common sense on the part of sampling personnel in discerning the difference between routine and extreme case scenarios. Sampling personnel must be alert to their surroundings (unusual circumstances, odors, presence of dead animal or plant life in the area, etc.) while:

- Collecting and recording visual and physical data
- Collecting soil samples, both surface and subsurface
- Adhering to procedures
- Maintaining the **Chain-of-Custody** (COC) Record
- Preserving program integrity

The **Quality Assurance Plan (QAP)** must define the uses of the data. The resulting **Field Sampling Plan (FSP)** will be based on existing knowledge of what chemicals may be present, the possible areas of contamination, and the possible migration of the chemicals through the soil. This existing knowledge can come from spill reports and preliminary surveys that identify discolored soil or distressed vegetation. Preliminary surveys may be performed to determine site lithology and identify the chemicals that pose the greatest hazard.

5.2.1 Background on Contamination of Soils. Soil contamination by elemental heavy metals such as lead or by chemicals such as pesticides that are relatively insoluble in water may be limited to the top few inches of soil. However, the area that is contaminated can be affected by soil erosion or by tracking or movement of the soil as a result of construction activities. Soil contamination by soluble metals such as plating wastes or by spills of concentrated organic chemicals such as gasoline, aviation fuel, solvents, transformer oil, etc. may extend to considerable depths and the concentration of the chemicals at any depth may not be easily predictable. Soluble metals and organic liquids are often carried through soil by percolating rain water, and the extent of movement of the contamination is affected by a number of factors including:

Solubility of the contaminant in water:

Some metals and most organic chemicals are soluble in water to the extent that the water becomes unfit for use as drinking water.

- Density of organic liquids:

  Light organic liquids such as gasoline, fuel, and oils will settle through soils which 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. The extent to which the underlying groundwater is contaminated then, is a function of the solubility of the organic liquid in the water.

  Dense, non-aqueous liquids, such as chlorinated solvents (trichloroethylene, tetrachloroethylene, carbon tetrachloride) or heavier chlorinated organic liquids such as chlorinated benzenes and 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:

Many organic chemicals can be degraded by micro-organisms in the soil. However, the micro-organisms require oxygen, and generally are not active at depths of more than a foot or so. Therefore, the surface soils may be relatively uncontaminated by organic chemicals because of biodegradation, while deeper soils may be quite contaminated because this process is not effective without oxygen.

- Type of soil (See **Figure 5-1**):

  Subsurface layers of clay are relatively impermeable to groundwater and organic liquids, but the clay particles can absorb some hazardous chemicals from the water resulting in increases in their concentration. Organic liquids may form pools in the low reaches of clay layers, or may flow down the surface of tilted clay layers.

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

  Porous non-organic soils such as sand or gravel allow for rapid movement of liquids (groundwater or spilled organic liquids). These soils do not absorb contaminants. For example, PCBs from spilled transformer fluid and from buried electrical capacitors have been found several hundred feet from the source of the contamination. Gasoline from leaks in underground storage tanks can be carried hundreds of feet by moving groundwater.

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

  The chemistry of the soil, such as its pH, can change the chemistry of the water percolating through it, which in turn affects the solubility of metals in the water. For instance, acidic rainwater can dissolve metals at the surface, but the metals may come out of solution and accumulate in the soil if the percolating water contacts a buried layer of lime.

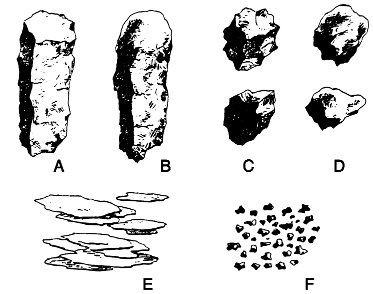


Figure 5-1 Types of Soil Structure:
A, prismatic; B, columner; C, angular blocky; D, subangular blocky; E, platy; F, granular.

- Presence of groundwater:

  Moving groundwater can carry hazardous contaminants for considerable distances (See **Figure 5-2**). 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. The contamination of groundwater with resultant damage to its usability as drinking water is one of the most serious effects of accidental spills and improper disposal of hazardous chemicals. A definition of a hazardous waste under RCRA is measured by determining whether its solubility in acidic water percolating through a municipal landfill will damage the drinkability of the underlying aquifer.

  An aquifer is a layer of soil or porous rock saturated with water. The aquifer surface confines vertical movement of organic liquids and percolating groundwater. Where the surface is exposed, as in stream valleys, contaminants in the groundwater can reappear as surface seeps.

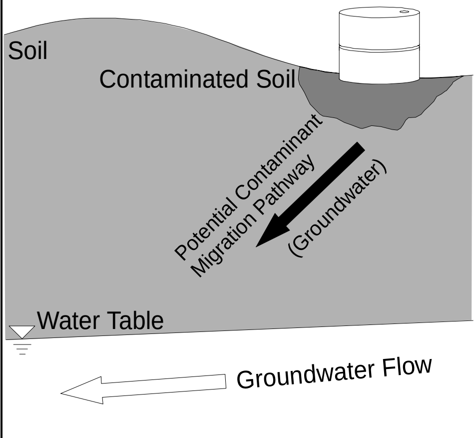


Figure 5-2 Contaminated Groundwater

Several different aquifers may be present at any given location, but may be separated by confining layers of clay or rock. If sampling holes penetrate the confining layer, contamination can move through the hole and contaminate deeper aquifers, resulting in increased environmental damage.

Alternatively, the deeper aquifers may be under hydrostatic pressure, and a sampling hole that pierces a confining layer may allow contaminated water from deeper aquifers to flow into shallower layers of soil.

5.2.2 Data Requirements. Ideally, a FSP should be based on knowledge of what contaminants are likely to be present and how the distribution of the contaminants may be affected by the lithology of the site. However, the purpose of the field work may be to define the lithology 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 for guiding subsequent work.

The most important data requirement from any field sample is the identification of exactly where the sample was collected. Stakes or flags should be installed to indicate the locations of soil samples. However, since sites will probably be disturbed by subsequent construction work, it is also 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/longitude location. For soil sampling programs, it is generally acceptable for the survey to define each sample location to an accuracy of one 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 second most important data requirement from any field sample is the collection of soil samples that represent field conditions *and* that 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 two inches and the soil should be sieved to eliminate stones larger than 2 millimeters 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 that must be excavated for proper disposal should not eliminate rocks, since the cleanup standards are based on the maximum allowable average concentration of PCBs in the remaining soil. The disposal requirements for PCB contaminated soil depend on when the spill occurred:
  - Soil contaminated with PCBs during 1987 or after is classified as hazardous based on the concentration of PCBs in the material spilled.
  - Soil contaminated with PCBs before 1987 is based on soil samples representative of the area to be excavated, including rocks, since the definition of PCB wastes is based on the average concentration of PCBs in the material.
- To determine whether excavated soil is classified as a hazardous material under RCRA, soil samples should be representative, including 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" 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 volatile organic chemicals 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.

The third data requirement is the geology and lithology of the site. Most permit applications for compliance require this information to be provided prior to starting any activities. Therefore the information may be referred to in the application documents. The following information should be noted or referenced to on a site map:

- Surface information:
  - Location of erosion patterns and accumulated runoff sediments.
  - Locations of possible sources of soil contamination, such as surface tanks, underground storage tanks, lagoons, waste pits, fire pits, or piles of trash or waste.
  - Locations of discolored soil.
  - Locations of areas covered by impermeable material, such as pavement and buildings.
  - Extent of areas of distressed vegetation.
  - Indications of geology and lithology, such as types of surface soil, exposed bedrock, standing water, and groundwater seeps.
- Subsurface information:
  - Type of soil as a function of depth at each sample location (layers of clay, peat, sand, silt, shale, etc.; layers having different colors; presence of large stones; etc.).
  - Moisture of soil at different depths, and depth at which the soil becomes saturated with water (i.e., depth to groundwater).

For some investigations of soil contamination by organic chemicals, field measurements may be made of the concentration of organic vapors in the air in bore holes as a function of depth, or next to newly exposed soil.

5.3 HAZARDS 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 soil sampling activities. Refer to **Section 3.3.3** and **Appendix D** for a specific hazard identification and appropriate reference for detailed control measures.

5.3.1 Environmental. Particularly during the initial site survey, the environment can be more hazardous than the toxic chemicals. Environmental hazards can include poison ivy, poisonous snakes, rabid animals, poisonous insects such as bees, wasps, and spiders, and the physical hazards of sunburn, heat exhaustion and frostbite. Safety precautions include a review of possible hazards before entering the site and the use of proper clothing and equipment.

Certain tasks such as site surveying and soil sampling at pre-determined locations may require that equipment be moved to inaccessible or unstable locations.

5.3.2 Safety

Some examples of safety concerns include:

- Problems associated with moving sampling equipment and containers across rough terrain either by hand carrying or driving off-road.
- Use of powered equipment such as power augers.

WARNING

- 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 back hoe to gain access to subsurface soil.
- Cave-in. Entry of personnel into pits more than four feet deep will require compliance with Occupational Safety and Health Administration (OSHA) shoring requirements to prevent injury from cave-ins.

5.3.3 Security. In extreme cases, two types of security issues should be addressed during the planning phases:

- Implement entry restrictions to prevent personnel exposure to toxic chemicals.
- Secure storage for sampling equipment and soil samples to prevent tampering.

Soil sampling is an intrusive activity that may expose sampling personnel to unidentified subsurface hazards.

5.3.4 Subsurface.

Some examples of subsurface hazards include:

- Buried munitions: Site records should be reviewed during the preparation of the **FSP**. If there is any chance of encountering buried munitions, 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 shall handle or sample explosive or suspected explosive materials. Certified explosives experts can be contacted at facilities listed in **Appendix G** of this manual.

WARNING

- 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 product 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.

5.3.5 Toxic Chemicals.

Collecting soil samples inherently involves the risk of exposure to hazardous chemicals. Major exposure scenarios that should be evaluated include:

- Skin contact with contaminated soil. The personnel who are involved in collecting the samples should wear disposable boot covers and rubber gloves as a minimum, and should consider the use of disposable Tyvek® coveralls to prevent contamination of clothing. The sample custodian should wear appropriate protective equipment 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. The personnel who are collecting the samples and decontaminating the sampling equipment should wear water proof rain gear or coated Tyvek® coveralls if there is a possibility of contact with contaminated water.

- Ingestion of contaminated soil. This usually occurs when food or drink is exposed to contaminated dust or is picked up with contaminated gloves. **No eating, smoking or chewing of gum or tobacco should be allowed 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 may also generate contaminated dust. Personnel should remain upwind of any source of dust. If the site is generally contaminated, full face air purifying respirators equipped with particulate filters should be worn.
- Inhalation of toxic vapors. Exposure of contaminated soil may generate toxic vapors from the evaporation of volatile organic chemicals. The HASP should establish air monitoring requirements for intrusive work, including specifying what air monitoring equipment is to 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 that may be present, the capabilities of the monitoring equipment, and the warning properties of the vapors.
- Confined space hazards. In general, pits more than four feet deep are considered to be confined spaces if there is no way for personnel to walk out. However, when digging into contaminated soil, there is a chance for the accumulation of flammable vapors or toxic gases including hydrogen cyanide and hydrogen sulfide. Any pit should be considered to be a confined space if there is any chance of generating toxic or flammable vapors from the soil and if one is required to put one's face below the top of the pit for any purpose, such as to examine soil conditions or collect soil samples. Normal confined space procedures shall be implemented, including air monitoring, the assignment of a confined space observer, and the possible use of respiratory protection.

5.3.6 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 shall participate in sampling activities. (See OPNAVINST 8023.2 for qualification and certification requirements.)

Special considerations are required when sampling explosive wastes which may be 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. General safety considerations include wearing personal protective equipment such as flameproof clothing, caps, safety goggles or face shields, conductive shoes and respirators where appropriate. Only non-sparking tools should be employed. 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, therefore, imperative that sampling personnel have a thorough knowledge of the characteristic dangers and safety requirements for individual explosive materials. (For safety and handling requirements refer to **NAVSEA OP5 VOLUME 1**, Ammunition and Explosives, Ashore Safety Regulations for Handling, Storing, Production, Renovation, and Shipping.)

WARNING

Only trained and certified explosive personnel shall handle or sample explosive materials. Certified explosive experts can be contacted at facilities listed in Appendix G of this manual.

5.4 PRINCIPLES OF SAMPLE COLLECTION.

The collection of useful information about soil contamination requires documentation of site surface conditions (locations of buildings, pavements, standing water, seeps, sediment runoff, discolored soil, etc.) and subsurface conditions (depth of various layers of soil, depth to groundwater, depth to bedrock at different locations). The chemical data obtained from analysis of soil samples is only useful within the context of site conditions, so it is very important that the exact location of each sample be documented and that the soil samples accurately represent the conditions at the site.

5.4.1 Preparation of Site Map. Documentation of site conditions and sample location requires a site map. A site map showing required sample locations should be included with the **FSP**. 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 rain water and other surface conditions that affect the movement of contaminated soil. If the FSP does not include a site map, one should be prepared by sampling personnel based on available facility drawings and field measurements.

5.4.1.1 **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.

5.4.1.2 **Surface Information.** Sampling personnel should validate the site map before starting sampling activities. Any discrepancies should be noted on the map, and the following surface information should be recorded if not already shown:

- Buildings
- Paved areas
- Unpaved roads and parking areas
- Surface areas of different types of soils, including fill areas (gravel roads, clay, sand, peat, etc.)
- Standing water (both permanent such as ponds and streams and temporary such as persistent puddles)
- Water seeps
- Water runoff patterns and accumulations of runoff sediments
- Exposed bedrock
- Discolored soil
- Stored materials, debris, soil piles, etc.
- Distressed (dead, wilted, or discolored) vegetation

It is recommended that photographs also be taken to document the site surface features. Photography requirements should be established by the Program Manager to ensure compliance with the policy and regulations of the facility.

5.4.1.3 **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 sample points. Depth of samples from the surface should be recorded. If it is necessary to sample more than a foot from the required location because of interferences such as 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 Field Log Book/Field Notes.

5.4.2 **Preliminary Tests and Observations.** A number of field tests and observations may be required to document sampling and subsurface conditions.

5.4.2.1 **Weather Conditions.** The weather conditions at the time of sample collection should be noted in the Field Log Book/Field Notes. 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, how recently) and freezing conditions (how cold, how deeply is the soil frozen).

5.4.2.2 **Description of Soil Types and Lithology.** The movement of groundwater and the transport of hazardous chemicals is strongly influenced by the presence and depths of different types of soil. Knowledge of subsurface lithology is necessary for an understanding of 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 the EPA *Description and Sampling of Contaminated Soils - A Field Pocket Guide*, is an excellent guide to the field description of soils. As necessary, the following soil features, when present, shall be described as a function of the depth from the surface:

- Color: soil colors should be determined with the use of a color chart (e.g., Munsell).
- 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 oil, gasoline, solvents or other organic liquids.
- Horizon thickness: layers of soil with distinct changes of the above features.

5.4.2.3 **Soil Gas.** Soil gas measurements, using field monitoring instruments such as photoionization detectors (PIDs) or flame ionization detectors (FIDs) can give an indication of the presence of volatile organic chemicals (VOCs) in the soil. The concentration of volatile organics measured with these instruments is not closely related to actual organic compound concentrations in the soil as the gas concentration depends on the following soil conditions:

- *Temperature* of the soil (higher temperatures increase the volatility of organic chemicals).
- *Moisture content* of the soil (organic chemicals vaporize faster from wet soils).
- *Absorption capacity* of the soil (organic contaminants vaporize slower from peat and other soils that contain significant organic material).
- *Instrument response factors* for the particular organic chemicals that are present in the air (both types of detectors respond differently to different chemicals).
- *Depth* of the bore hole and time that it has been open (organic vapors can sink to the bottom of an open bore hole, increasing the concentration at the bottom).

However, the following techniques can obtain at least qualitative information on the presence of VOCs in soil.

5.4.2.3.1 Down Hole. This technique measures the concentration of the organic vapors that have accumulated in the air in a bore hole. It is subject to all of the variables listed above. Stick the intake port of the instrument into the bore hole and record the maximum response. If the instrument has a sample pump, connect a sampling tube to the intake port and lower the tube down the bore hole, recording the maximum instrument response.

5.4.2.3.2 Sniff. This technique measures the concentration of VOCs in air directly adjacent to freshly exposed soil. In addition to the variables listed above, the measured levels of organics will depend on the porosity of the soil and the wind velocity. Break open a soil sample and immediately hold the intake port of the instrument close to the newly exposed surface; record the maximum instrument response.

5.4.2.3.3 Head Space. The technique used should control moisture content, temperature and the tendency for organic vapors to settle. Use of this technique should be reviewed by a chemist to ensure that the instrument and controlled temperature will measure the contaminants suspected. Blanks and background measurements should be made to aid with the evaluation of the final results. In some methods, water or a nonchlorinated solvent is added to the container. The sealed container is gently shaken and may be placed in the sun, water bath, or controlled temperature device and heated to a known temperature. After a stated period of time, a probe to detect organic vapors is inserted into the headspace of the container and the maximum instrument reading is recorded.

5.4.3 Access to Soil. 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 all cases, care should be taken to avoid buried utilities.

5.4.3.1 Paved Areas. It may be necessary to use a jackhammer to remove pavement from a sampling location; provisions should be made for replacing the pavement after the sampling is complete.

5.4.3.2 Grass Areas. If the sampling location is covered with grass 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 at the conclusion of sampling. The stainless steel shovel should be decontaminated between sampling locations. Turf can also be removed by hand from sample locations provided the person has donned a pair of clean nitrile or PVC gloves.

5.4.3.3 Surface Samples. Soil samples should be of undisturbed soil, and not material that is temporarily lying on the ground. Unless the FSP specifically requires the sampling of surface debris, remove organic debris (leaves, etc.) and accumulated trash (paper, cans, bottles, demolition rubble) to expose the soil. The FSP should define the depth of surface samples. Specific state and local compliance programs may define surface samples as the first three, first six, or first twelve inches below the vegetation layer. Note in the Field Log Book/Field Notes how much and what kind of material was removed.

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

5.4.3.4.1 Test Pit. 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. Samples should be collected from undisturbed material from the walls or bottom of the pit, not from soil that is excavated from the pit.

Advantages:

- 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.

Disadvantages:

- Safety:

  The backhoe may have to be completely 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 requires a person's nose to be below the level of the surface should be considered a confined space entry, and all air monitoring and use of personnel protective equipment (PPE) requirements must be complied with.

  WARNING

  Excavation of contaminated soil can lead to the vaporization of significant amounts of toxic chemicals, or generation of considerable contaminated dust, resulting in an inhalation hazard to the sampling personnel. The FSP should address this potential hazard.

- Environmental:

  If soil contamination is suspected, a large amount 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 at a greater depth or contaminated soil from subsurface layers may be exposed on the surface.

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.

5.4.3.4.2 **<u>Augers</u>.** An auger is a hole making tool that is screwed into the soil (See **Figures 5-3 and 5-4**). In general, augers should only be used 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. In addition, when augers are lowered into bore holes, 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.

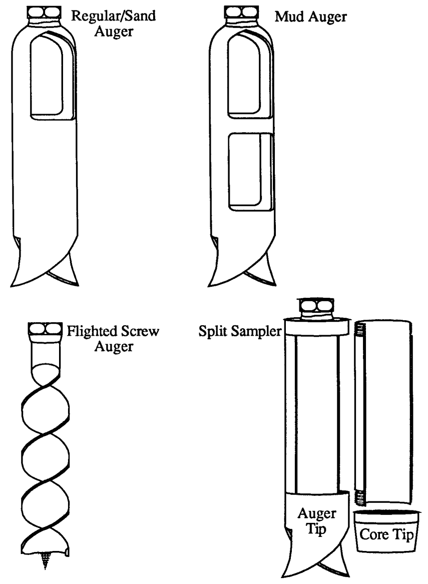


Figure 5-3 Augers

A hand auger can be used to expose soil as deep as four 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.

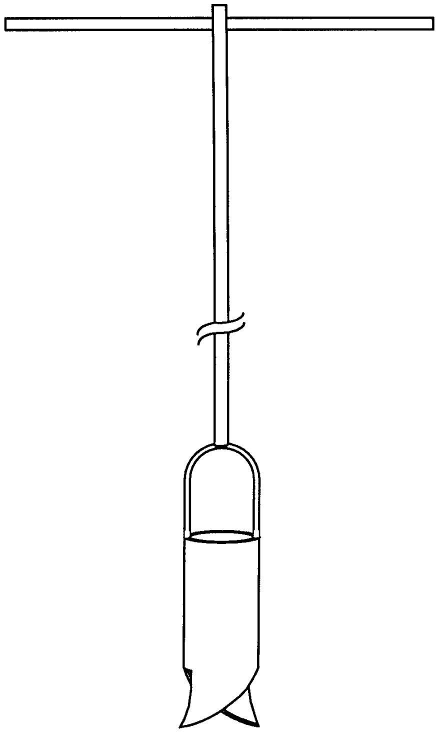


Figure 5-4 Bucket Auger

Various types of augers are available. The major types, their advantages, and their limitations are summarized in **Table 5-1**. A typical auger consists of a handle, a stainless steel extension rod, and a bit. Additional extensions can be added for deeper holes. Place the auger over the desired sampling location and turn the handle while exerting downward pressure. The turning motion of the handle will rotate the bit down into the soil. To remove the auger, turn the handle in the opposite direction; the soil will remain in the bit and can be removed by using a clean stainless steel scoop or spoon.

<u>Auger Use Procedures:</u>

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 to 6 inches of surface soil from an area one 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 accumulated soils to prevent accidentally brushing loose material back down the bore hole when removing the auger or adding drill rods. Shovel the loose soil onto a piece of plastic sheeting.

Table 5-1 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 (marches)	
In Situ Soil Recovery Auger	Collection of soil sample in reusable liners; closed top reduces contamin- ation 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	

4. After reaching the desired depth, slowly and carefully remove the auger from the bore hole.
1. Remove the auger from the drill rods and replace with a precleaned tube sampler. Install 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.

5.4.3.4.3 Drill Rig. 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 purposes of classification. Disadvantages of using a drill rig include increased labor (the drilling crew) and equipment rental costs.

NOTE:
Drill rigs may be required in examining hazardous waste sites, but generally they would not be used in routine sampling operations.

5.4.4 Use of Soil Sampling Equipment. Soil sampling equipment should be selected according to the objectives of the sampling activity. It is important to determine the depth at which samples will be collected prior to sampling visit. Sample depth will determine the types of sampling equipment required. All sampling equipment shall be decontaminated prior to use at each sampling location. It is preferable that this be done in a controlled area before entering the site. Field decontamination of equipment between sampling locations shall be done as described in **Section 5.4.7**.

5.4.4.1 Trowel, 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 are also used to mix and handle soil obtained from deeper locations. All stainless steel scoops, spoons, and tulip bulb planters should be decontaminated and wrapped in aluminum foil prior to use. If the equipment is not pre-wrapped in aluminum foil or other clean wrap material, sampling personnel should assume that it is not clean and should not use it.

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; these are unacceptable because the plating can peel off and get into the soil sample. Stainless steel scoops are preferred. However, scoops made from other materials may be acceptable in certain instances. The decision for equipment construction of material other than stainless steel will be made by the Program Manager or designee. Stainless steel trowels and scoops can be purchased from scientific or forestry equipment supply houses. Stainless steel spoons can be purchased in housewares departments.

5.4.4.2 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 5-2**.

5.4.4.2.1 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. A standard 2-foot split spoon is advanced ahead of the auger with a 140 pound hammer. Because of 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.

Table 5-2 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	

Split Spoon Sampler Use Procedures:

1. Use an auger or drill rig to open a bore hole 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 over the bore hole in a perpendicular position.

4. Drive the tube utilizing a drop hammer, sledge hammer or well rig if available. Do not drive past the bottom of the head piece as this will result in compression of the sample.
5. Record the number of blows required to advance the sampler each 6 inch increment.
6. Carefully pull the sampler out of the hole. Open the sampler by unscrewing the drive shoe and head and split the barrel. Discard the top two or three inches of soil. If split samples are desired, a decontaminated stainless steel knife should be used to divide the tube contents in half verticall
7. For samples to be analyzed for volatile organics, transfer the sample directly into clean volatile organic analyte (VOA) vials, minimizing exposure to air. (VOA vials are special wide mouthed bottles with lids that have a thin Teflon® liner under a silicone septum. VOA vials are used for samples that will be analyzed for volatile organic analytes.) For samples to be analyzed for other parameters such as semivolatile organics or metals, place soil into a stainless steel bowl and mix it thoroughly using a stainless steel scoop or trowel; place homogenized soil into the required sample containers.

5.4.4.2.2 Shelby Tube Samplers. A Shelby tube consists of a thin walled tube with a tapered cutting head. This 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 geologic information but may be used in obtaining samples for chemical analysis.

Advantages:

- Easily cleaned
- Samples to be analyzed for volatile organic chemicals may be sent to the laboratory in the tube, minimizing air exposure

Disadvantages:

- Difficult to drive into hard soils
- Not durable in rocky soils
- Sometimes difficult to extract soil samples from the tube

Shelby Tube Sampler Use Procedures:

1. Use an auger or drill rig to open a bore hole down to the depth to be sampled.
2. Place the sampler over the bore hole 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 sit for a few minutes to allow soils to expand in the tube.
5. Before pulling out the tube, rotate the tube at least two revolutions to shear off the sample at the bottom.
6. If the sample is to be shipped for further geologic analysis, the tube must be appropriately prepared for shipment. Generally this is accomplished by sealing the ends of the tube with wax in order to preserve the moisture content. In such instances, the procedures and preparation for shipment shall be in accordance with ASTM D1586-83.
7. For samples to be analyzed for volatile organics, extrude the soil directly into VOA containers to minimize the exposure of the samples to air. Alternatively, the sampling tube can be sent to the laboratory. To ship a full sampling tube, cover the ends of the tube with clean aluminum foil, held in place by rubber bands or wire ties; then clean the outside of the tube, apply a sample container label to the tube, and place it in a resealable clear plastic food bag.
8. For samples to be analyzed for non-volatile contaminants, extrude each sample into a stainless steel bowl and mix thoroughly using a stainless steel scoop or trowel; place the mixed soil into the required sample containers.

5.4.4.2.3 Hydraulic Ram. Truck mounted hydraulic ram soil samplers gain access to subsurface soil by pushing a closed-end 3/4" diameter pipe into the soil, down to the sampling depth. The pipe contains a removable sampling tube, and is capped with a retractable penetrating point. After the pipe is pushed to the desired depth, the penetrating point is retracted and the pipe is advanced to collect the soil sample inside the removable tube. This device can reach depths of 12 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:

Advantages:

- Retrieves relatively undisturbed soil cores up to 18" long. By collecting cores from different depths at closely adjacent locations, it is possible to develop a continuous core for soil classification purposes.
- Rapid - one core sample can be collected every 10 minutes.
- Minimizes the release of organic vapors, reducing personnel exposure hazards.
- Does not generate any waste soil that must be stored as potentially hazardous waste.

Disadvantages:

- Requires the rental of special equipment and trained operators.
- Soil cores are only 1/2" in diameter, so several core samples may have to be obtained from a single sampling location to meet the laboratory sample quantity requirements.

5.4.4.2.4 **Veihmeyer Sampler.** The Veihmeyer sampler is recommended for core sampling of most types of soil. It may not be applicable to sampling stony, rocky, or very wet soil.

This sampler was developed by Professor F. J. Veihmeyer of the University of California at Davis. The parts of a basic sampler are given as follows and the basic sampler is shown in **Figure 5-5**:

a. Tube, 1.5 m (5 ft.)
b. Tube, 3 m (10 ft.)
c. Drive Head
d. Tip
e. Drop hammer, 6.8 kg (15 lb.)
f. Puller jack and grip*

* recommended for deep soil sampling.

Advantages:

- Can achieve substantial depths with appropriate length of tubing
- Various heads available for different soil types

Disadvantages:

- Very difficult to clean
- Parts needed for sampler are not appropriate for certain analyses

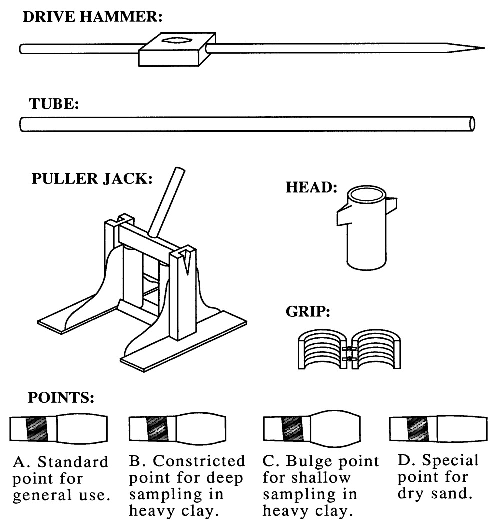


Figure 5-5 Veihmeyer Sampler

Veihmeyer Sampler Use Procedures:

1. Assemble the sampler by screwing in the tip and the drive head on the sampling tube.
2. Insert the tapered handle (drive guide) of the drive hammer through the drive head.
1. Place the sampler in a perpendicular position onthe material to be sampled.
2. With one hand holding the tube, drive the sampler into the material to the desired sampling depth by the pounding drive head with the drive hammer. Do not drive the tube further than the top of the hammer's guide.
3. Record the length of the tube that penetrated the material being sampled and the number of blows required to obtain this depth.
4. 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.
5. Rotate the sampler at least two revolutions to shear off the sample at the bottom.
6. Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head and rotate about 90°.
7. 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.

8. 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.
9. Transfer sample into laboratory cleaned sample bottles and follow procedures for preservation and transport (See **Section 4.7** of this manual).

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.

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

5.4.5.1 **Sieving.** Sieving eliminates large rock fragments. It may be appropriate where EPA protocols require that only fine soil material be analyzed. Refer to **Figure 5-6** 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:

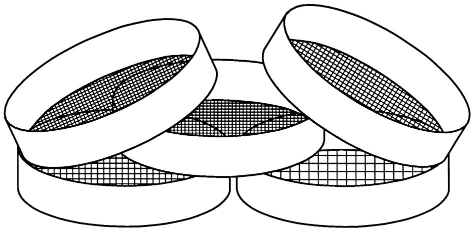


Figure 5-6 Sieves

Break up soil aggregates and pull apart vegetation and root mat, if present. Weigh non-soil vegetation fraction, and archive or discard, as required by the FSP.

Remove large rocks and weigh. Archive for possible analysis.

Crush the entire soil sample with a rolling pin, stainless steel spoon, or some similar tool. Mix thoroughly with stainless steel spoon.

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 must be classified as hazardous waste under RCRA.

  Use disposable stainless steel screen for samples to be analyzed for semivolatile organic contamination.

  Use Teflon® screens for samples to be analyzed for metal contamination.

Spread out the sample, mark off quarters, and take samples from each quarter in a consecutive manner until appropriate sample volume is collected. Archive remaining sample for future analysis, if specified.

Before shipping the samples to the laboratory, shake each sample container to mix thoroughly.

5.4.5.2 Split Samples. Material from a single sampling event, made homogeneous and divided into separate samples for submission to different laboratories.

5.4.5.2.1 Volatile Organic Analysis. For splitting samples to analyze for volatile organic chemicals, collect two core samples from adjacent locations. Alternatively, split a soil core longitudinally with a clean knife and place one half of the core in 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 must be uniform to ensure data comparability.

5.4.5.2.2 Semivolatile Organic and Metal Analysis. Place soil in a clean stainless steel bowl. Crush and thoroughly mix the soil with a clean stainless steel spoon. One method of sample selection is to form the soil into a cone and divide the cone in half vertically. Fill each set of sample containers with the soil from one half of the pile. The method used for sample mixing and placing in the sample container should be documented and uniform to ensure data comparability.

5.4.5.3 Composite Samples. Composite samples are only to be collected after full justification and documented rationale are presented in the sampling plan. Composite sample collection techniques should not be used if justified solely on the basis of reducing the sample testing cost. The method of compositing the sample should be documented and uniform to ensure future data comparability. The method for splitting the sample either for compositing of purposes of analysis should be the same for both field operations and the laboratory.

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 field sampling plan 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 and a portion of the material is collected and combined with other positive areas. In most cases, equal amounts of material are collected and mixed in the sampling container. Protocols for combining the sample are based on the site conditions, the parameters being measured, the regulatory limit, and the data quality objectives. The rationale for the use of glass, stainless steel and polyethylene equipment for combining samples, and the rationale for combining the soil in equal amounts, proportional amounts, or other amounts should be documented. An example of collecting a sample for detection of petroleum products using a composite sampling technique is to use an appropriately-sized, disposable, recloseable freezer storage bag. Collect a minimum of three equally sized samples (ten is preferred) from designated sample locations and place in the sample bag. Close and secure the bag using care to eliminate trapped air. Mix the sample by tumbling and kneading until well mixed. Separate sample(s) into appropriate containers.

NOTE:

> Soil samples collected for determination of volatile organic chemicals should never be composited or mixed.

5.4.6 Restoration of Sample Locations. Sample locations are to be restored to grade so that they do not result in a tripping hazard. Precautions should be taken to prevent introducing contaminated soil into uncontaminated soil horizons, or leaving contaminated soil exposed on an otherwise clean surface.

5.4.6.1 Backfill Sample Holes. Soil pits and bore holes will be backfilled after samples have been collected. For shallow pits and bore holes where only one soil horizon is involved, it may be allowable to backfill with the material from the pit or hole. This is to be determined by the Program Manager and specified in the **FSP**.

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

5.4.6.2 Grouting Bore Holes. Bore holes that penetrate a confining layer (silt, clay or rock that acts as the base of an aquifer) must be grouted to prevent migration of contaminated water between aquifers. Grouting consists of filling the bore hole with concrete or bentonite slurry as specified by the Program Manager in the FSP. Details of the grouting (depth of bore hole, depth to confining layer, mix ratio of the grout and amount used) should be recorded in the Field Log Book for each bore hole.

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

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

5.4.7 Decontamination of Sampling Equipment. 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 must be decontaminated before being used to prevent contaminants from being carried over from one sample to the next. If sampling equipment must be reused, it must be decontaminated prior to sampling and between each sample location.

5.4.7.1 Decontamination of Sampling Equipment. All field decontamination must take place on a decontamination pad lined with plastic. Solvent rinse liquids must be collected in dedicated containers. All aqueous decontamination liquids must be stored in Department of Transportation (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 FSP must detail proper waste material handling procedures.

5.4.7.2 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.

5.4.7.2.1 Organic. Cleaning and decontamination procedures for equipment used to collect soil samples for **analysis for organic chemicals** are as follows. Decontamination must be carried out over separate containers to separate used water and solvents. All liquid wastes from equipment decontamination procedures shall be collected in appropriate DOT approved compatible containers for storage and possible disposal as hazardous waste.

1. Remove heavy soil deposits with high pressure water or steam.
2. Wash equipment with water and laboratory grade glassware detergent.
3. Rinse generously with tap water.
4. Rinse with distilled water.
5. Rinse with acetone, hexane or biodegradable organic cleaner.

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 to rinse the equipment for submission to the laboratory as an equipment decontamination bank.

6. Air dry.
7. Wrap trowels and other small sampling tools in aluminum foil if they are not used immediately after being decontaminated.

8. Information concerning decontamination methodology, date, time, and personnel should be recorded in the Field Log Book/Field Notes.

5.4.7.2.2 **Inorganic.** The cleaning procedures for field sampling equipment used to collect soil samples for **metals analysis** are as follows. Decontamination must be carried out over a container to catch used water and solvents. All liquid wastes from equipment decontamination procedures shall be collected in DOT approved compatible containers for storage and possible disposal as hazardous waste. Add one half cup of lime to each drum to neutralize the nitric acid.

1. Remove heavy soil deposits with high pressure water or steam.
2. Wash equipment with water and laboratory grade glassware detergent.
3. Rinse generously with tap water.
4. Rinse with distilled or deionized water.
5. For low level contamination or where equipment decontamination 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).

NOTE:

Personnel protective equipment recommended for handling nitric acid includes nitrile rubber gloves, boot covers, rubber apron or coated Tyvek® coveralls, and eye protection such as face shields or goggles.

6. Rinse with distilled or deionized water.

NOTE:

At least once each day, collect the rinse water as an equipment decontamination blank.

7. Air dry.
8. Wrap the sampling equipment with aluminum foil unless it will be used immediately.
9. Information concerning decontamination methodology, date, time, and personnel should be recorded in the Field Log Book/Field Notes.

5.4.7.2.3 **Both Organic and Inorganic.** The following procedures should be used to decontaminate field sampling equipment that will be used to collect a soil sample to be analyzed for both metal and organic chemicals. Aqueous and organic liquid wastes from equipment decontamination procedures shall be collected in DOT approved compatible containers for storage and possible disposal as hazardous waste. Add one half cup of lime to each aqueous waste drum to neutralize the nitric acid.

1. Remove heavy soil deposits with high pressure water or steam.
2. Wash equipment with water and laboratory grade glassware detergent.
3. Rinse generously with tap water.
4. Rinse with distilled water.
5. For low level contaminates or if equipment decontamination blanks are contaminated, rinse with acetone, hexane or biodegradable organic cleaner.

NOTE:

All substitutes must be approved by the Program Manager. At least once each day collect samples of the solvent and water as an equipment decontamination blank.

6. For low level metal contaminates or if equipment decontamination blanks are contaminated, rinse stainless steel equipment with 10% nitric acid (trace metal or higher grade HNO_3 diluted with distilled or deionized water).

NOTE:

At least once a day, collect the rinse water as an equipment decontamination blank.

7. Rinse with distilled or deionized water.
8. Air dry.
9. Wrap sampling equipment with aluminum foil unless the equipment will be used immediately.
10. Information concerning decontamination methodology, date, time, and personnel should be recorded in the Field Log Book/Field Notes.

5.4.7.3 **Preparation of Equipment Decontamination Blanks.** Equipment decontamination blanks provide a check on the cleanliness of sampling equipment and the purity of the water or solvent used for the final rinse(s). The purpose of equipment decontamination blanks is discussed in detail in **Section 4.7.7.3**.

At least one equipment decontamination blank should be submitted each day for each type of rinse solvent (water, acetone, etc.) that is used. The FSP should specify the size and material of each sample bottle required for equipment decontamination blanks, and should also specify any required preservatives for these liquid samples.

Equipment decontamination 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. Detailed packaging, labeling and shipping instructions are presented in **Appendix F**.

5.4.8 Waste Material Storage and Disposal. All material generated during soil sampling projects must 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 personnel protective equipment.

5.4.8.1 Soil Cuttings. Soil cuttings from bore holes 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 bore holes or test pits unless specifically authorized by the FSP or regulatory authority.

Soil cutting 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 personal protective equipment.

5.4.8.2 Used Disposable Personal Protective Equipment. All disposable solid contaminated equipment (plastic sheets, screens, coveralls, boot covers, gloves, etc.) should be placed in plastic bags for temporary storage and sealed in metal barrels for final storage, transport and disposal based on generator knowledge or test results.

5.5 SOIL SAMPLING PROCEDURES. The procedures described here generally apply to any type of soil sampling. Departures from procedures contained in the FSP must be documented and justified.

5.5.1 Preparation. Thorough preparation is the key to a successful field sampling program. Necessary steps include:

- Review the FSP and safety plan or HASP (as appropriate to the scope of the project) to identify special equipment and procedures.
- Coordinate access to the sampling location with facility management, including facility security, industrial hygiene, respirator program, and confined space program personnel as relevant to the planned work. Coordinate with the facility to ensure the availability of emergency response personnel if needed and to ensure the proper disposal of hazardous wastes generated during sampling. Establish communication requirements and procure communication equipment required to ensure access to emergency response services.
- Coordinate with the laboratory to ensure that someone will be available to receive the samples when they are delivered, and to ensure that the laboratory can meet the required holding time requirements given the prior commitments of the lab to other programs.
- Obtain the required equipment. An example of a standard list of sampling equipment for soil samples is listed at the end of the chapter.
- Inspect the site to ensure that present conditions are the same as indicated in the FSP and that all designated sample locations are accessible.
- Survey site as necessary to locate defined sampling points.
- Set decontamination facilities for personnel and equipment as required by the HASP.
- Set up and mark required Exclusion Zone(s) and Contamination Reduction Zone(s) and establish required site security when necessary.

5.5.2 Sample Collection. Field sampling personnel should be familiar with all of the technical issues and documentation requirements discussed in **Chapters 3, 4, and 5** of this manual. All samples shall be collected using the procedures specified in the **FSP**. The following sampling procedures can be used when there are no special conditions present or special requirements specified by the FSP.

5.5.2.1 Surface Samples. The simplest and most direct method of collecting surface soil samples for subsequent analysis is with a spade and scoop. Very accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sampler. A flat, pointed trowel can be used to cut a block of soil two inches deep. Chrome-plated tools, common with garden implements such as potting trowels, should be avoided.

Sampling Procedure

1. Clear the area to be sampled of any surface debris (twigs, rocks, litter). Cut grass down to the level of the soil and remove.
2. Define a sample area such that a two 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 precleaned stainless steel trowel. Place the soil in a clean stainless steel bowl.
5. Remove all roots and other debris, rocks and pebbles. Describe the amount and kind of material that is removed in the Field Log Book.
6. If instructed by the FSP, sieve the sample as described in **Section 5.4.5.1**. Crush the soil sample and screen the soil through a 2 mm mesh stainless steel (for organic analysis) or Teflon® (for metals analysis) sieve. Note in the Field Log book the amount, size and type of material that does not

pass the sieve and is discarded. If the soil is too wet and cohesive to pass through the sieve, note in the Field Log Book that the required sieving was not done and enter this notation of the Field Sampling Form. Inform the Program Manager that the sample was not sieveable.

7. Fill the required sample containers.
8. Label the sample containers, placing one part of the sample label on the Field Sampling Form and the large part of the label in the Field Log Book.
9. Complete Field Log Book documentation.
10. Pack samples for shipping and complete **COC** Record for each shipping container.
11. Send (or FAX) completed Field Sampling Forms and **COC** Records as specified in the FSP.
12. Deliver samples to the laboratory or to the courier service for overnight delivery to the laboratory.

5.5.2.2 <u>Subsurface Samples</u>. Hand-held augers and thin-walled tube samplers can be used separately or in combination. 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 to 2 meters can be readily sampled and up to 6 meters where conditions are favorable. A drill rig should be used to gain access where deeper samples are required or where soil conditions are not favorable for the use of augers. **Tables 5-1** and **5-2** summarize the advantages and disadvantages of different types of augers and tube samplers for sampling under different soil conditions.

Specific sampling tools may require slightly different handling methods. For example, if sampling devices and drill rod extensions do not have quick connect fittings, crescent or pipe wrenches may be required to change equipment configurations. The procedure described below is for hand-held equipment. Procedures for **power-driven augers** or **tube samplers** are essentially the same.

1. Attach the auger bit to a drill rod extension and attach the "T" handle to the drill rod.
2. Clear the area to be sampled of any surface debris (twigs, rocks, litter). It may be advisable to remove the first 8 to 15 cm of surface soil for an area approximately 15 cm in radius around the drilling location to prevent near-surface soil particles from falling down the hole.
3. Begin drilling, periodically removing accumulated soils. This prevents accidentally brushing loose material back down the bore hole when removing the auger or adding drill rods.
4. After reaching the desired depth, slowly and carefully remove auger from boring.

For **thin-walled tube samplers**:

1. Remove auger tip from drill rods and replace with a precleaned thin-walled tube sampler. Install 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 thin-walled tube sampler).
2. Carefully lower corer down bore hole. Gradually force corer into soil. Care should be taken to avoid scraping the bore hole sides. Hammering of the drill rods to facilitate coring should be avoided, as the vibrations may cause the bore walls to collapse.
3. For samples to be analyzed for volatile organics: if a sufficient number of tube samplers are available, seal the ends of the sampler with aluminum foil, mark the top and bottom ends of the sample, put a sample container label on the sampling tube, and put the tube into a resealable clear plastic food bag. Submit the sampling tube to the laboratory.
4. For samples collected for analysis for parameters other than volatile organics or if a sufficient number of tube samplers are not available or if the FSP specifies that the samples are to be submitted to the laboratory in glass containers, remove corer and unscrew drill rods. Remove core from device (this may require removing cutting tip) and discard top of core (approximately 2.5 cm), to eliminate soil that may have fallen down from higher horizons.

For **split-spoon samplers**:

1. 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. Place the sampler over the bore hole in a perpendicular position.
2. Drive the tube utilizing a drop hammer, sledge hammer or well rig if available. Do not drive past the bottom of the head piece as this will result in compression of the sample. Record the number of blows required to advance the sampler each 6 inch increment.
3. Withdraw the sampler and open by unscrewing the drive shoe and head and splitting barrel. Discard the top two or three inches of soil. If Split samples are desired, a decontaminated stainless steel knife should be used to divide the tube contents in half longitudinally.

5.5.2.2.1 <u>Volatile Organic Chemicals</u>. Tube samplers are preferred when collecting for volatiles. Soil samples should be taken from auger cuttings only if soil conditions make collection of undisturbed cores impossible. Soil recovery probes with dedicated or reusable liners (see **Table 5-2**), will minimize contact of the sample with the atmosphere.

1. Record in the **Field Log Book/Field Notes** the depth from the surface to the top and the bottom of the core.
2. Fill sample containers as follows:
   - It is preferable to submit the soil samples in the sampling tubes.
   - Where samples must be submitted to the laboratory in glass containers, use the first adequate soil core to fill a 120 mL septum vial or in a glass wide mouth jar with a Teflon®-lined cap, maintaining and handling the sample in as undisturbed a state as possible. Do not mix or sieve soil samples. Ensure the sample containers are filled to the top to minimize volatile loss. Secure the cap tightly.
   - If Field Duplicate samples are required, it is preferable to collect separate samples from adjacent locations. If this is not possible, split the core vertically before filling the sample jars.
3. Examine the hole from which the sample was taken with an organic vapor instrument after each sample increment. Record any instrument readings.
4. Label and tag sample containers, and record appropriate data in Field Log Book/Field Notes (depth, location, etc.).
5. Place glass sample containers in sealable plastic bags, if required, and place containers in iced shipping container. Samples should be cooled to 4°C as soon as possible.
6. Complete **COC** Records and deliver the samples to the laboratory as soon as possible to minimize sample holding time (see **Appendix H** for maximum holding times for various constituents).
7. Follow specified decontamination and disposal procedures.

5.5.2.2.2 <u>Other Parameters</u>. For each sample, approximately one liter of soil will be placed into a clean stainless steel bowl. This soil will be homogenized by mixing with a clean trowel. Sample container jars will be filled with portions of this soil as required for Test samples, Field Duplicate samples and Matrix Spike/Matrix Spike Duplicate samples.

1. Record in the **Field Log Book** the depth from the surface to the top and the bottom of the core.
2. Describe the soil color, type, etc. in the Field Log Book. If the sample consists of two different types of soil (sand and clay, different color layers, etc.) 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 Field Log Book/Field Notes.
3. Mix the sample in a stainless steel, aluminum (not suitable when testing for aluminum), or glass mixing container using the appropriate tool (stainless steel spoon, trowel, or pestle).
4. If required by the FSP, sieve the soils through a mesh screen of the specified size. Use a precleaned stainless steel screen for semivolatiles, or Teflon® lined screen for metals (some metals in stainless steel could contaminate the sample).
5. Divide the screened soil into quarters, and fill each sample container with portions of soil from each quarter. Separate sample containers may be required for semivolatiles, metals, Duplicate samples, Field Duplicate samples, Split samples, and Matrix Spike/Matrix Spike Duplicate samples.
6. Secure the cap tightly. The chemical preservation of soils is generally not required.
7. Label sample containers, and record appropriate data in the Field Log Book/Field Notes (depth, location, other observations).
8. Place glass sample containers in sealable plastic bags. Place containers in an iced shipping container. Samples should be cooled to 4° C as soon as possible.
9. Complete **COC** Records and ship the samples to the laboratory as soon as possible to minimize sample holding time (see **Appendix H** for maximum holding times for various constituents). Schedule arrival time at the analytical laboratory to give as much holding time as possible for scheduling of sample analyses.
10. Follow required decontamination and disposal procedures.

<u>5.6 SAMPLING EQUIPMENT LIST</u>. Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to soil sampling:

Auger, Drill Rig
Split spoon sampler, and other tube sampler
Trowel, Scoops, Spoons
Hammer to advance split spoon
Plastic sheeting (under coring spoils)
Shovel (to refill sampling holes)
Steam jenny to clean auger and split spoons
Stainless steel sampling trowel
Stainless steel mixing bowl
Detergent
Acetone, hexane or biodegradable organic cleaner (Optional)
Nitric acid (Optional)
Tap water
Distilled or Deionized water (ASTM Type II)
Sample jars for soil
Sample bottles for water (equipment decontamination blanks)
Storage/disposal container for wash water, acid and solvents
Lime to neutralize nitric acid wastes (Optional)
Storage/disposal container for soil cuttings
Grouting materials such as concrete, bentonite slurry
Camera and film to document sampling process (Optional)
Tape to measure sample depth
Waterproof pens
3-part sample container labels
Clear wide plastic tape to cover sample container labels and custody seals
Shipping container and materials including:
Bubble wrap
Clear plastic resealable freezer food bags for sample containers, ice and **COC** Record
Vermiculite
Ice
Disposal drum for used PPE, plastic sheeting
Miscellaneous field test kits for analyte(s) (optional)

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 6

AQUATIC SEDIMENT SAMPLING

6.1 PURPOSE. This chapter provides procedures to secure sediment from an identifiable water location for chemical analysis and ecological toxicity studies.

6.2 SCOPE. The scope of work covers sediment sampling on site and includes methods used to collect adequate sediment samples.

6.3 HAZARDS AND SAFETY PRECAUTIONS. See **Chapter 3, Section 3.3.3**.

6.4 PREPARATION. Sediment samples will be collected to determine whether discharge from the site significantly impacts surface water bodies and sediments near the site. A potentially more serious and common problem associated with surface water is contamination of sediments. Therefore, it is important to monitor sediment concentration if it is suspected that surface water has been contaminated. The choice of sampling locations for sediments is similar to the criteria applied to surface water sampling. Field-screening techniques can be used in defining areas of contamination at the site. However, it should be noted that sediment contamination often consists of inorganics and/or non-volatile organics, for which field-screening techniques are limited. Therefore, in designing a sampling protocol, consideration of the contaminants of concern is very important. Prior to conducting field sediment sampling a preliminary on-site investigation should be conducted to determine the contamination of concern which affects human health and the environment at the site.

The preparation of a sampling program addresses the initial evaluation of data from the preliminary site investigation and background information collected during the sampling process, including the following:

- An analysis and summary of the site background and the physical setting of sediments at the site
- Determination of the impact of the site on sediments (e.g., from surface run-off and seeps)
- Determination of the contaminants concentration in upstream samples
- Determination of the contaminants concentration in downstream samples
- Determination of the waste characteristics of sediments
- Determination of the extent and volume of contamination
- Evaluation of the preliminary assessment of human health and environmental impact; and additional data needed to conduct the baseline risk assessment for the site
- Determination of potential erosion of the site contributed by sediment build-up

Much of the above information can be obtained through record searches, preliminary site investigation (phase I investigation), and agencies such as the **United States Geological Society (USGS)** and **Soil Conservation Service (SCS)** and other public agencies. Field investigation and sampling should be conducted in accordance with references from local state or Corps of Engineers offices.

Prior to initiating any field and sampling activity, sampling personnel should review and discuss in detail the safety plan or HASP (as appropriate to scope of project). All monitoring and protective equipment should be checked thoroughly at this time. Prior to collecting sediment at each location, all water monitoring equipment should be thoroughly rinsed with ASTM Type II or better quality water.

Recommended field equipment for sediment sampling is listed in **Section 6.7**.

Prior to initiating sediment sampling, record the following information in the Field Log Book/Field Notes:

- Sample location, ID number, date and time
- The depth of sediment and physical characterization of the sediment should be noted

6.4.1 Criteria for Sediment Sampling. An evaluation of drainage patterns of the site indicates the optimal sediment sampling locations. One sediment sample should be collected where run-off sediment accumulates and other sediment samples collected where overflow enters the stream channel near the drainage or leachate seeps. The background sample should be collected at the upstream location of the site. Additional sampling may be required to assess seasonal/tidal fluctuation and multiple point discharges. Data needs for evaluating sediment contamination may be quite extensive depending on the depth and extent of potential contamination.

6.4.2 Sampling Design. This section outlines the basic statistical design available for use in the sampling of any environmental media. Information obtained from the study should be representative of the sampling system in the study. The sampling design must provide a desirable type of information, with maximum reliability and minimum cost. One technique which can help reduce the effects of variation in sampling is to divide the sampling area into smaller, more homogenous sub-areas called strata. These strata are defined by some identifiable boundary, based on chemical and physical properties of the sample.

Environmental pollution behavior often is difficult to understand without some graphical means of displaying the spatial relationship of data. Use of a grid helps ensure sampling coverage for portions of the entire study area, rather than at only certain randomly selected points. The systematic sample plan refines the statistical design, whose purpose is to identify the extent of contamination from discharges or non-point sources. This efficiency may result from either obtaining a smaller sampling error with the same number of samples or from reducing the number of sample units required to produce a specified error. Once the number of samples is determined, their locations can be planned. Here, the grid becomes the basis for selecting sample locations. The systematic sampling plan is an attempt to provide better coverage of the environmental study to show the horizontal and vertical extent of the contamination present at the site. In systematic sampling, samples are collected in a regular pattern, usually a grid or a line transect over the areas under investigation. The orientation of the grid should be such that the lines in one direction are parallel to the general trace of the suspected potential contamination at the site. The spacing of the grid is also important to determine and delineate the extent of contamination at the site.

CAUTION

> Compositing of a number of subsamples is another technique often used to average and reduce the effects of variation in the results. One consideration when compositing is to review the potential for "hot spot" dilution by compositing from a variety of sites. This dilution may reduce the reported concentration for any area and must be taken into consideration as part of the data quality objectives criteria.

6.4.3 Sediment Sampling. Objectives of sediment sampling are as follows:

- Determine the impact of point source discharges on sediments.
- Develop site specific sediments transport mechanism for permit applications such as dredging.
- Determine contaminant concentration upstream and downstream of non-point or point source discharges.
- Determine the complexity of exposure pathways to aquatic/terrestrial life (including the complexity of release sources and transport media).

For a summary of sediment sampling and sampling considerations from the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) Program, refer to **Tables 6-1 and 6-2**.

6.4.3.1 Wetland Sediment Sampling. Many Naval sites have been built on or adjacent to natural wetlands. Any activity may affect the wetland habitat and may become contaminated by inflow of leachate or run-off from the site. Anaerobic sediments in the wetland may concentrate and sequester heavy metals or complex organics present in the leachate.

When dredging for sediment sampling below the water surface, the type of equipment depends on considerations such as the need to control secondary contamination migration, depth of the contaminated sediment, and depth of the excavation of the sediment at the wetland.

6.4.3.2 Semi-Solid and Fluid-Like Sediment Sampling. Semi-solid sediments are composed of saturated earth or other materials. These materials may flow when disturbed. For shallow water deposits, waders and an Ekman dredge generally will procure an adequate sample. If waders are insufficient, a flat-bottomed boat should be substituted.

6.5 SAMPLE COLLECTION PROCEDURES.

Factors contributing to the selection of a sediment sampler include the width, depth, flow and bed characteristics of the media to be sampled.When sampling from a boat, ensure that the free end fo the rope is firmly attached to the boat so that the sampling will not be lost if the rope slips from your hands.

6.5.1 Sampling Equipment Procedures.

6.5.1.1 Veihmeyer Sampler. The Veihmeyer sampler (see **Chapter 5, Section 5.4.4.2.4**) is recommended for core sampling of most types of soil. It may not be useful for sampling stony, rocky, or very wet soil.

This sampler was developed by Professor F. J. Veihmeyer. The parts of a basic sampler are given as follows:

- Tube, 1.5 m (5 ft.)
- Tube, 3 m (10 ft.)
- Drive Head
- Tip, Type A, General use [a]
- Drop hammer, 6.8 kg (15 lb.)
- Puller jack and grip [b]

[a] Only one of each part is needed

[b] Recommended for deep soil sampling

Advantages:

- Can achieve substantial depths with appropriate length of tubing
- Various heads available for different soil types

Disadvantages:

- Very difficult to clean
- Parts needed for sampler are not appropriate for certain analyses

Veihmeyer Sampler Use Procedures:

1. Assemble the sampler by screwing in the tip and the drive head on 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 on 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 drop hammer. Do not drive the tube further than the tip of the hammer's guide.
5. Record the length of the tube which 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°.
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; carefully recover the sample from the tube and place in an appropriate sampler container.
11. Transfer sample into laboratory cleaned sample bottles and follow procedures for preservation and transport (see **Section 6.6**).

6.5.1.2 **Ekman Dredge.** Sediment samples in lakes, ponds, and other calm waters may be collected with an Ekman dredge, although the physical composition of the bottom determines to a great extent the type of sampler that must be used to collect an adequate sample. The Ekman dredge consists of a square box of sheet stainless steel 6 x 6 inches in cross-section (See **Figure 6-1**).

The lower opening of this box is closed by a pair of strong jaws so made and installed that they oppose each other.

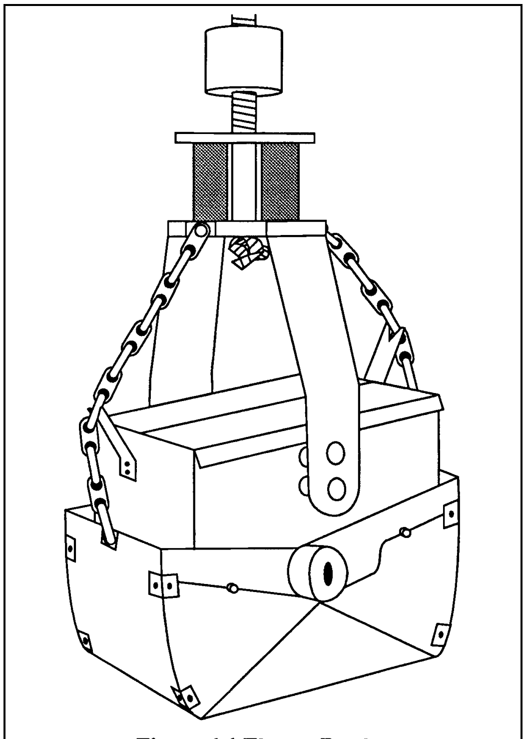

Figure 6-1 Ekman Dredge

When open, the jaws are pulled apart so that the whole bottom of the box is open. The jaws are held open by chains attached to trip pins. To close the dredge, the trip pins are released by a brass messenger sent down the attachment rope and the jaws snap shut by two strong external springs. The hinged top of the box is equipped with a permanent 30-mesh screen to prevent loss of sample if the sampler sinks into sediment deeper than its own height. The sampler is especially adapted for use in soft, finely divided sediment. It does not function properly on sand bottoms or hard substrates.

Advantages:

- Very light weight
- Samples top 6 inches of soft sludge or sediment
- Easy to operate and to recover sample

Disadvantages:

- Jaws tend not to close in sand, gravel, or sticks

Ekman Dredge Use Procedures:

1. Open dredge by pulling up on the chains and hooking them over trip pins.

WARNING

Ensure that fingers are not where they may be caught by the jaws of the dredge. The jaws snap shut with great force.

2. If in a boat, tie the free end of the rope to the boat.
3. While holding on to the stainless steel messenger, lower the dredge into the water.
4. When the dredge settles into the sediment, keep the rope taught and release the stainless steel messenger.
5. When an audible sound indicates that the messenger has hit the dredge, slowly bring the dredge to the surface and place over an empty bucket.
6. Pull up on the chains and release the dredge contents into the bucket.

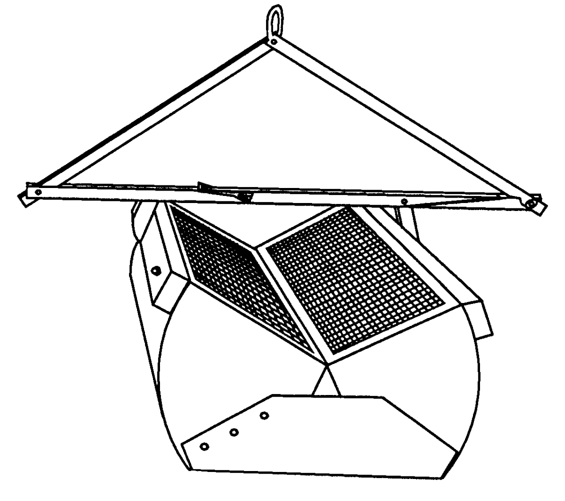

Figure 6-2 Ponar Dredge

7. Collect a suitable sample from the dredge contents.

6.5.1.3 Ponar Dredge. The Ponar Dredge is a clam shell-type scoop activated by a counter-lever system (See **Figure 6-2**).

The shell is opened and latched in place and slowly lowered to the bottom. When tension is released on the lowering cable, the latch releases and the lifting action of the cable on the lever system closes the clamshell.

Ponars are capable of sampling most types of sludges and sediments, from silts to granular material. They are available in a "petite" version with a 232 square centimeter sample area, light enough to be operated without a winch or crane. Penetration depths will usually not exceed several centimeters. Grab samplers, unlike corers, are not capable of collecting undisturbed samples. As a result, material in the first centimeter of sludge cannot be separated from that at lower depths. The sampling action of these devices causes agitation currents which may temporarily resuspend some settled solids. This disturbance can be minimized by slowly lowering the sampler the last half meter and allowing a very slow contact with the bottom. Collection of sludge or sediment samples must be done after all overlying water samples have been obtained.

Advantages:

- Ability to sample most types of sludges and sediments, from silts to granular material
- Light weight
- Large sample can be obtained intact, permitting further intervals

Disadvantages:

- Shock wave from descent may disturb fine sediments, from silts to granular material
- Not capable of collecting undisturbed samples
- Can lose possible contaminants when pulling samples through water column
- Incomplete closure of jaws can result in sample loss

Ponar Dredge Use Procedures:

1. Attach a decontaminated Ponar to the necessary length of sample line.
2. Measure and mark the distance to bottom on the sample line. A secondary mark, 1 meter shallower, will indicate proximity so that lowering rate can be reduced, thus preventing unnecessary bottom disturbance.
3. Open sampler jaws until latched. From this point on, support sampler by its lift line or the sampler will be tripped and the jaws will close.
4. Tie free end of sample line to fixed support to prevent accidental loss of sampler.
5. Begin lowering the sampler until the proximity mark is reached.
6. Slow rate of descent through last meter until contact is felt.
7. Allow sample line to slack several centimeters. In strong currents more slack may be necessary to release mechanism.
8. Slowly raise dredge clear of surface.
9. Drain excess liquid through screen.
10. Place dredge into a stainless steel or Teflon® tray and open.

11. Collect a suitable aliquot with stainless steel spoon or equivalent and place into the appropriate sample container. Care should be taken to collect material which has not contracted the dredges's sides.
12. Transfer sample into laboratory cleaned sample bottles and follow procedures for preservation and transport (see **Appendix H**).

6.5.1.4 **PACS Sludge Getter.** The PACS Sludge Getter can be used in very viscous material to collect a representative sample at depth (See **Figure 6-3**).

The Sludge Getter is an extra heavy duty grab sampler for heavy sludge areas. Its massive 32 lb. weight and conical bottom allows penetration of extremely viscous material. Constructed of type 316 stainless steel, it has a cup capacity of approximately 1,000 mL.

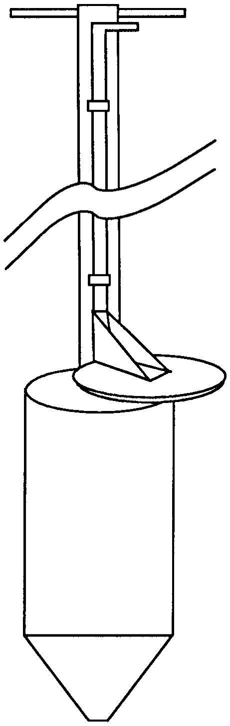

Figure 6-3 PACS Sludge Getter

Advantages:

- Can be used in heavy sludge
- Can collect discrete samples at depth
- Bag liner can be used with sampler
- Easily decontaminated with steam cleaner or solvent wash

Disadvantages:

- Heavy

PACS Sludge Getter Use Procedures:

1. Cover the sample vessel with the lid by maneuvering the handle on the sampler.
4. Lower the sampler vessel to the desired depth.
5. Uncover the sampler vessel using the handle, and allow the sample vessel to fill.

6. After the vessel has had time to fill, slide the lid back into place with the handle.
7. Remove sampling device from sludge.
8. Transfer sample into laboratory cleaned sample bottles and follow procedures for preservation and transport (see **Appendix H**).

6.5.1.5 **PACS Grab Sampler.** The PACS Grab Sampler can be used to collect sludge samples from ponds, lagoons and containers (see **Chapter 7, Section 7.5.1.7**). Grab samples can be obtained at discrete depths.

For sludge sampling, the PACS Grab Sampler is available with a wide necked bottle and large openings to allow the sample to enter the bottle. The sampler consists of a 1,000 mL bottle that screws onto the end of the 6 ft. long handle. The control valve is operated from the top of the handle once the sampler is at the desired depth.

Advantages:

- Allows discrete samples to be taken at depth

Disadvantages:

- Depth of sampling is limited by length of pole
- Not useful in very viscous sludges
- Hard to decontaminate

PACS Grab Sampler Use Procedures:

1. Assemble the sampler in accordance with manufacturer instructions.
2. Operate sampler several times to ensure proper adjustment, tightness of the cap, etc.
3. Submerge sampler into sludge to be sampled.
4. When desired depth is reached, open sample bottle.
5. Retrieve sampler.
6. Transfer sample into laboratory cleaned sample bottles and follow procedures for preservation and transport (see **Appendix H**).

6.5.1.6 **Hand Corer.** This device is essentially the same type of thin-walled corer which is used for collecting soil samples (See **Figure 6-4**).

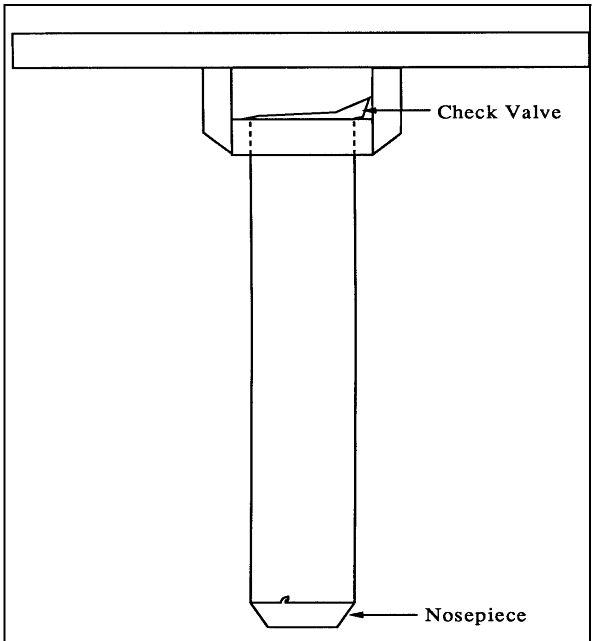

Figure 6-4 Hand Corer

The corer is modified by the addition of a handle to facilitate driving the core, and a check valve on top to prevent wash out during retrieval through an overlying water layer. Hand corers are more applicable to sludges but can be used for sediments provided the water is very shallow (a few centimeters). It should be noted, however, that this method can be disruptive to the water/sediment interface and might cause significant alterations in sample integrity if extreme care is not taken.

Some hand corers can be fitted with extension handles which allow collection of samples underlying a shallow layer of liquid. Most corers can be adapted to hold liners.

Advantages:

- Easy to use
- Minimal risk of contamination

Disadvantages:

- Can disrupt water/sediment interface
- Does not work well in sandy sediments

Hand Corer Use Procedures:

1. Decontaminate prior to use.
2. Force corer in with a smooth, continuous motion.
3. Twist corer and withdraw in one motion.
4. Remove nosepiece and withdraw sample.
5. Transfer sample into an appropriate sample bottle with a stainless steel spoon or equivalent.
6. Transfer sample into laboratory cleaned sample bottles and follow procedures for preservation and transport (see **Appendix H**).

6.5.1.7 **Gravity Corer.** A gravity corer is a metal tube with a replaceable tapered nosepiece on the bottom and a ball or other type of check valve on the top (See **Figure 6-5**). The check valve allows water to pass through the corer on descent but prevents washout during recovery. The tapered nosepiece facilities cutting and reduces core disturbance during penetration.

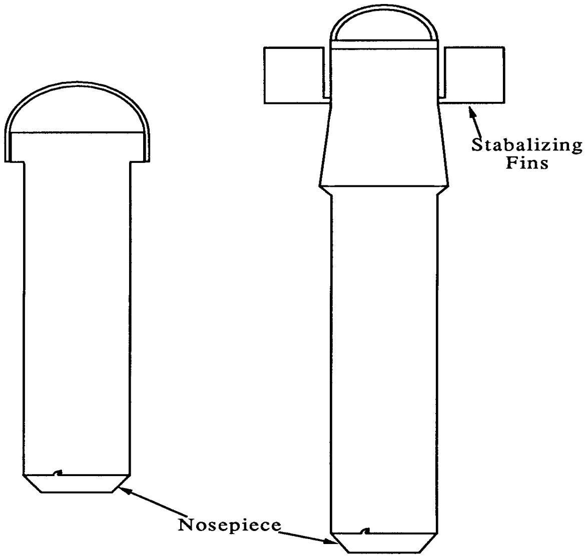

Figure 6-5 Gravity Corers

Corers are capable of collecting samples of most sludges and sediments. They collect essentially undisturbed samples which represent the profile of strata which may develop in sediments and sludges during variations in the deposition process. Depending on the density of the substrate and the weight of the corer, penetration to depths of 75 cm (30 in) can be attained.

CAUTION

Care should be exercised when using gravity corers in vessels or lagoons that have liners since penetration depths could exceed that of substrate and result in damage to the liner material.

Advantages:

- Collects undisturbed samples

- Core samples transferred in liner

Disadvantages:

- May damage liners in vessels or lagoons

Gravity Corer Use Procedures:

1. Attach decontaminated corer to the required length of sample line.
2. Secure the free end of the line to a fixed support to prevent accidental loss of the corer.
3. Allow corer to free fall through liquid to bottom.
4. Retrieve corer with a smooth, continuous lifting motion. Do not bump corer as this may result in some sample loss.
5. Remove nosepiece from corer and slide sample out of corer into stainless steel or PTFE (e.g. Teflon®).
6. Transfer sample into appropriate sample bottle with a stainless steel lab spoon or equivalent.
7. Follow procedures for preservation and transport (see **Appendix H**).
8. Decontaminate before use at next location.

6.5.2 Collection Procedures.

6.5.2.1 Volatile Organic Compounds (VOCs). Use the following procedures for collecting samples for VOCs:

1. Set up a table or work bench on the shoreline (any appropriate location) to extract the soil from the coring tube and fill the necessary containers.
2. Approach the sampling location from downstream.
3. Advance the coring tube 2-feet into any sediment contained location.

NOTE:

Wetland, stream, or other sediments samples should be collected in this manner.

4. Carefully remove the coring tube with the sample and hand it to on-shore personnel.
5. Extract the sediments from the coring tube.
6. Remove the cap from a 40-mL septum (Teflon®-faced silicon rubber) vial. Avoid contact with the inner surface.
7. Inspect the vial for air bubbles. If air bubbles are present, discard the vial and start with step 1.
8. Attached a number and tag to the vial, seal it in a resealable bag, and place it into a cooler with ice. Place sufficient ice bags in the cooler to completely surrounded the samples and to maintain a temperature of 4°C until the samples are received by the laboratory.
9. Record all appropriate data in Field Log Book/Field Notes.

6.5.2.2 Sediment Sampling for Base Neutral Acids/ Pesticides/PCBs / Other Organics / Toxicity Characteristic Leaching Procedure (TCLP). Follow steps 1 through 5 for VOCs in **Section 6.5.2.1** of this manual.

For Base Neutral Acids, Pesticides, PCBs and other Organics:

Fill the 8 oz. glass bottle with sediments from the coring tube. No preservative should be added for this sample when samples are semisolid or solid. Liquid sediment samples may need to be chemically preserved to maintain analyte integrity.

For TCLP:

1. Fill two additional 16 oz. glass bottles using the same procedure as above.
2. Attach a number label and tag to the vial, seal it in a resealable bag, and place it into a cooler with ice. Place sufficient ice bags in cooler to completely surrounded the samples and to maintain a temperature of 4°C until the samples are received by the laboratory. Add no preservative to TCLP sam-ples unless required by the FSP.
3. Record all appropriate data in the Field Log Book/Field Notes.

6.5.2.3 Sediments Sampling for Metals/Inorganics. Follow the sampling procedures in **Section 6.5.2.2** for metals and inorganic analysis using the same container for sediment sample collection.

6.5.2.4 Grain Size Distribution Samples. According to ASTM C316 methods, grain size sediment samples should be collected for grain size analysis in the same manner that all other samples are collected as referred to above. The grain size distribution is necessary to determine the sediment's physical characterization at particular locations. This test determines whether sediments are plastic or non-plastic in nature. The grain size distribution also determines the penetration resistance of the sediments by rigid objects, which helps in evaluating the compaction factor of the sediments. The bulk density can be determined with this parameter as to how much excavation occurred during the remedial action at the site. The grain size distribution also determines the particle capacity to adsorb inorganic contaminants. Samples should be collected for this analysis in an 8 oz. glass jar, with no added preservative added. Attach a label and tag to the jar, seal it in a resealable bag and place it into a cooler with sufficient ice to maintain temperature of 4°C until the samples are received by the laboratory. Record all appropriate data in Field Log Book/Field Notes.

6.5.2.5 Sediment Engineering Parameters and Properties. Texture, clay content, and strength behavior are the key properties of sediments. These properties are directly connected with the chemical properties of sediments to adsorb the organic and inorganic contamination at the site. The **Atterburg Limits** define various states of fine-grained sediments material ranging from dry to liquid. The **Shrinkage Limit** is the point at which a further reduction in water does not cause a decrease in the volume of the mass. The **Plastic Limit** is the point at the which soil changes from a semi-solid to a plastic state due to the water content. At the plastic limit, a fine-grained soil will just begin to crumble when rolled into a thread approximately 3 mm in diameter. This physical parameter is necessary to determine what actions are necessary if excavation of the sediments are required to clean up the site.

6.6 QUALITY ASSURANCE / QUALITY CONTROL (QA/QC).

The following protocol should be used to ensure the integrity and accuracy of data collected during sediment sampling. All samples must be accompanied by a completed **Chain-of-Custody** (COC) Record to verify the integrity of the sediment samples. Field Blanks, Trip Blanks, Equipment Decon-tamination Blanks and Field Duplicates should be collected to enable QA evaluation of data accuracy. Any field decontamination process must be documented. Follow pre-approved procedures to ensure the quality of the sampling.

Final data must be reviewed for correctness of numerical input, numerical calculations, and validity of the equation used. Each site QA/QC varies with the degree and nature of contamination, and site location. The following are the main elements of Quality Assurance Plan (QAP) for sediment sampling:

- QA objective for measurements
- Sampling Procedures
- Sample custody
- Calibration procedures
- Analytical Procedures
- Data reduction and calculations
- Internal quality control
- Performance audit
- Data assessment or validation
- Quality assurance report
- Corrective actions

Two Field Duplicate samples and Field Equipment Blank samples should be collected and analyzed. Matrix spikes and matrix spike duplicates may be performed one time per matrix to validate the method selected with the matrix being analyzed. More or less frequent QC may be included in the QAP of the site specific Field Sampling Plan (FSP).

Data Quality Objectives (DQOs). The collection of data requires that sampling and analysis procedures be conducted with properly operated and calibrated, if necessary, equipment by trained personnel. The precision is defined as the degree of mutual agreement among individual measurements with an accepted reference or true value. Dredging permits and discharge applications may address more or less extensive program requirement. The documented DQOs should discuss the rationale for the sampling and testing, the expected precision, bias, comparability and the methods used for accomplishing the sampling and testing operations.

For guidance, refer to the USEPA"s *Conducting Remedial Investigation/Feasibility Studies for CERCLA Municipal Landfill sites*, EPA/540/p-91/001, February 1991. A summary of sediment sampling requirements at different locations appears in **Tables 6-1** and **6-2**. These are presented as guidance, but are not requirements for compliance monitoring.

6.7 SAMPLING EQUIPMENT LIST.

Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to sediment sampling:

Safety equipment, as required
Veihmeyer Sampler
PACS Sludge Getter
Hand Corer
Gravity Corer
Preservation chemicals for liquid sediments

pH meter, Dissolved oxygen (DO) meter, and thermometer
HNU photoionization detector (if necessary for personal protection)
Stainless steel measuring tape
Trip blank samples when measuring volatile organics
Equipment cleaning materials and reagents
Decontamination detergent
Ekman dredge
Coring tube
Ponar dredge
gallon pail
Small boat, if necessary
Camera, if necessary

Table 6-1 Summary of Aquatic Sediment Sampling

Media to be Investigated	Sample Locations	Minimum number of samples
WETLANDS AND OTHER SENSITIVE AREAS: Collect sediment and observe and collect aquatic life	Non-affected areas and affected area	Three from non-affected areas; three to five from affected area, depending on relative size

Table 6-2 Sediment Sampling Considerations

Location	Sampling location	Consideration
RIVERS	Upstream; at site; downstream	Minimum of three samples at each location; sampling locations should be at least one mile apart
INTERMITTENT STREAMS	Affected area only	Three samples from random locations
PONDS	Deepest area; inlet area; outlet area; area of obvious affect; each discernible bay	One sample each area
LAKES	Deepest area; inlet area; outlet area; area of obvious affect; each discernible bay	Relative size of waterbody should determine number of sample collections; minimum of one sample at each location

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 7

SURFACE WATER SAMPLING

7.1 PURPOSE. This chapter provides procedures for surface water sampling. Surface water samples are collected and analyzed to characterize surface water quality and/or determine pollutant concentrations.

The role of sampling personnel is critical to the water sampling program relative to:

- Collection of visual and physical field data.
- Adherence to sampling and preservation procedures.
- Maintenance of the **Chain-of-Custody (COC)** Record.
- Integrity of the program.
- Sampling personnel must be properly trained in sample collection.

7.2 SCOPE. This chapter provides guidance for:

- Surface water sampling.
- Identifying and evaluating the potential impact of a discharge on a body of water.
- Determining the type and extent of contamination in nearby surface water, which may adversely affect the human environment or drinking water supplies.
- Evaluating the impact of the discharge on sensitive environments (e.g., habitats, wildlife).
- Determining contaminant concentration upstream and downstream of the discharge.

7.3 HAZARDS AND SAFETY PRECAUTIONS. See **Chapter 3, Section 3.3.3**.

7.4 PREPARATION. Surface water samples are collected to determine whether discharge from the site has a significant impact on the surface water body. A preliminary surface water quality survey should measure pH, temperature, and dissolved oxygen at the points along the shorelines, wetlands, creeks, and ponds. Surface water sampling should be based on visual evidence of seepage or discharge streams. Sampling points should be established at the locations where distinct changes of pH, temperature, conductivity or dissolved oxygen indicate the possible presence of contamination or leachate discharge.

CAUTION

Approach each sample location from downstream, being careful to minimize disturbing any sediments which might become entrapped in the sample.

Field equipment that should be available at the time of sampling is listed at the end of this chapter.

Prior to sampling at each location, the water monitoring probes should be rinsed thoroughly with ASTM Type II or better quality water. Record the following information in the **Field Log Book/ Field Notes**:

- Sample location, ID number, date, water temperature at time of collection.
- pH, dissolved oxygen (DO), conductivity, and depth of water at sample collection location along with any equipment calibration data.
- Water depth at midpoint, if applicable.
- Description of flow rate, velocity, weather condition at time of sampling, and physical characteristics of sample.

7.4.1 Surface Water Runoff. Many sites are near bodies of surface water including rivers, intermittent streams, shorelines, ponds, and lakes. The following might contaminate the surface water body:

- Site surface water runoff.
- Surface seepage of leachate.
- Leachate seepage to groundwater, which recharges to a surface water body.
- National Pollutant Discharge Elimination System (NPDES) permitted discharge to waterway.

Surface water runoff investigations should be coordinated with groundwater, leachate runoff, and soil investigations. Rationale for the locations of surface water sampling and monitoring are derived from the investigations of other media, visual observations of vegetation or biota, and permit application requirements.Surface water investigations determine:

- The impact of the discharge on surface water.
- Contaminant concentration in upstream and downstream samples.
- Characterization of surface water runoff.

Much of the above information can be obtained through a record search, initial site investigation, and from agencies such as the **United States Geological Survey (USGS)** or the **Soil Conservation Service (SCS)**. Field sampling should be conducted in accordance with EPA, Corps of Engineers, State, or local government compliance requirements and guidelines. Surface water runoff samples should be collected upstream and downstream of the site when the need for changes in water quality are being assessed. 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 is based on the data quality objectives for data interpretation and assessment. Specific sampling locations and procedures should be identified in the sampling plan.

Additional sampling locations might be considered, depending upon the size of the site, number of streams or rivers near the site, and location of natural drainage scales 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 USGS, various state agencies and public water supplies monitor river/stream flow and water quality at various points along major rivers or streams. Resulting data can be used for water levels, flow rate, drainage information, and water quality needs. Precipitation data can be acquired from local weather bureaus or the National Climatic Data Center in Asheville, North Carolina.

Some sites are located near intermittent streams. These streams often transport contamination from the site as a result of surface water runoff during or after a period of heavy rainfall. If contamination is suspected as a result of seasonal runoff, surface water samples should be collected during and/or immediately following periods of heavy rainfall. An evaluation of the optimal sampling locations should be made. The EPA Stormwater program provides guidance on possible sampling programs for determining the concentration of contaminants during rainfall events. **It is particularly important to sample the stormflow runoff from the first 30 minutes of significant flow, or by permit requirements.** One sample should be collected where runoff or overflow enter 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 estimation of water drainage in particular areas.

7.4.2 Leachate. Prior to initiating any field actions, the sampling personnel should review and discuss, in detail, the **HASP** and procedures. 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 collection locations should be identified for sampling which include the outfalls to shorelines. The location of leachate discharge ultimately depends on site 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 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 a dedicated precleaned container should be used to collect the surface water 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, Total Organic Carbon (TOC), chloride, nitrate, phosphate, ammonia, and sulfide. All samples should be analyzed according to the permit requirements.

7.4.3 Wetland Sampling. Some sites have been built on or next to natural wetlands or other sensitive environments. The contamination can migrate from the site through surface water to wetlands which adsorb heavy metals and complex organics, impacting the wetlands. Wetlands should be defined in accordance with the *Federal Manual for Identifying and Delineating Jurisdictional Wetland* (The US Fish and Wildlife service et al., 1989).

Sampling of wetlands for chemical and biota studies is 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 Field Sampling Plan (FSP). The FSP will describe the procedures to meet the data quality objectives for interpreting the environmental impact of suspected contamination.

7.5 SAMPLING PROCEDURES. Prior to initiating any field and sampling activity, sampling personnel should review and discuss in detail the safety plan or HASP (as appropriate to scope of project). All monitoring instruments and personnel protective equipment (PPE) should be checked thoroughly at this time. Prior to sampling at each location, the water monitoring equipment probes should be rinsed thoroughly with ASTM Type II or better quality water. At minimum, surface water samples should be collected near drainage or leachate seeps.

Sampling of surface water 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 vary to best fit each situation. The most important goal of surface water sampling is to collect the sample representative of all the horizons or phases present in the liquid.

Specific regulations for grab and composite sampling are defined differently on the federal and state level. A Grab Sample is an individual sample of at least 100 milliliters collected at a randomly-selected time over a period of not exceeding 15 minutes. A composite sample is a combination of at least 8 sample aliquots of at least 100 milliliters, collected at periodic intervals during the operating hours of a facilities over a 24 hour period. The composite must be flow proportional; either the time interval between each aliquot or 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. For GC/MS Volatile Organic Analysis (VOA), aliquots must be combined in the laboratory immediately before analysis. Four (4) rather than eight aliquots or grab samples should be collected for VOA. These four samples should be collected during actual hours of discharge over a 24 hour period and need not be flow proportioned. Only one analysis is required. Since state and local regulations must be at least as stringent as the federal regulations, many states and localities possess more stringent regulations. For example, South Carolina's sampling regulation defines grab and composite sampling more stringently than the Federal definition. The State specifies that grab samples are individual discrete or single influent or effluent portion of at least 100 mL collected at a time representative of the discharge.

Composite samples are defined as being one of the following:

- An influent or effluent portion collected continuously over a specified period of time at a rate proportional to the flow.
- A combination of not less than 8 influent or effluent grab samples collected at regular (equal) intervals over a specified period of time, properly preserved, and composited by increasing the volume of each aliquot in proportion to flow. If continuous flow measurement is not used to composite in proportion to flow, the following method will be used: take an instantaneous flow measurement each time a grab sample is collected. At the end of the sampling period, sum the instantaneous flow measurements to obtain a total flow to determine the partial amount (percentage) of each grab sample to be combined to obtain the composite sample.
- A combination of not less than 8 influent or effluent grab samples of equal volume but at variable time intervals that are inversely proportional to the volume of the flow. That is, the time interval between aliquots is reduced as the volume of flow increases.
- A combination of not less than 8 influent or effluent grab samples of constant (equal) volume collected at regular (equal) time intervals over a specified period of time, while being properly preserved.
- Continuous flow or the sum of instantaneous flows measured and averaged for the specified compositing time period shall be used with composite sample results to calculate quantity. It is therefore imperative to check state and local regulations before conducting surface water sampling for regulatory compliance.

7.5.1 Operation of Sample Collection Devices.

7.5.1.1 Laboratory Cleaned Sample Bottle. The most widely used method for collecting surface water samples is simple immersion of a laboratory cleaned sample bottle or certified precleaned bottle. Using the sample bottle for actual sampling eliminates the need for other equipment. This method also reduces the risk of introducing other variables into the sampling event.

Advantages:

- Easy operation
- No field decontamination necessary

- No other equipment needed

Disadvantages:

- Outside of bottle comes in contact with sample
- Labeling may be difficult
- Is not possible when bottles are pre-preserved
- Sample collected from top of surface water depending on bottle filling technique

Sample Bottle Use Procedures:

1. Make sure bottles are intact, with proper fitting lids.

CAUTION

> Collect samples for volatile organics analysis first to prevent loss of volatiles due to disturbance of the water.

2. Immerse bottle into surface water and allow water to run slowly into bottle until full to zero headspace.
3. Follow preservation procedures.
4. Transport sample to laboratory after proper **Quality Assurance/Quality Control (QA/QC)** actions (See **Section 7.6**).

7.5.1.2 Pond Sampler. The pond sampler is used to collect liquid waste samples from disposal ponds, pits, lagoons, and similar reservoirs (See **Figure 7-1**). It consists of an adjustable clamp attached to the end of a two or three piece telescoping aluminum tube serving as the handle. The clamp is used to secure a sampling beaker. Though commercially available, the sampler is easily and inexpensively fabricated. Tubes can be readily purchased from most hardware or swimming pool supply stores. The adjustable clamp and sampling beaker (stainless steel or Polytetrafluoroethylene) can be obtained from most laboratory supply houses.

Advantages:

- Relatively inexpensive to make
- Can sample depths or distances up to 3.5m

Disadvantages:

- Difficult to obtain representative samples in stratified liquids
- Difficult to decontaminate when viscous liquids are encountered

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.

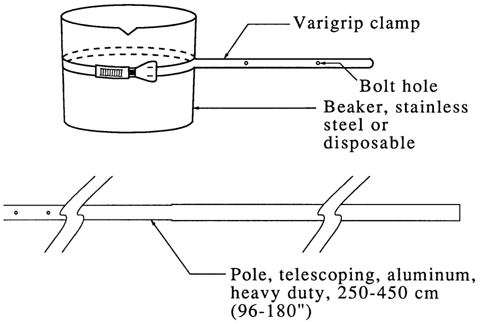

Figure 7-1 Pond Sampler

Pond Sampler Use Procedures:

1. Assemble the sampler. Make sure that the sampling beaker or sample bottle and the bolts and nuts securing the clamp to the pole are tightened properly.
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/device edge.
5. Empty the sampler slowly, allowing the sample stream to flow gently down the side of the bottle with minimal entry turbulence. When applicable, always fill VOA vials first, to zero headspace.
6. Repeat steps 1-5 until a sufficient volume is drawn.
7. Follow preservation procedures.
8. Transport samples to the laboratory after proper **QA/QC** actions (See **Section 7.6**).
9. Dismantle the sampler and store in plastic bags for subsequent decontamination.

7.5.1.3 Weighted Bottle Sampler. The weighted bottle sampler can be used to sample liquids in storage tanks, wells, sumps, or other reservoirs that cannot be adequately sampled with another device (See **Figure 7-2**). 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 during sampling. There are a few variations of this sampler.

However, the preferred one is that recommended in ASTM procedures, which uses a stainless steel or carbon steel bottle basket that also serves as the weight sinker. The weighted bottle sampler can either be fabricated or purchased.

Advantages:

- Sampler remains unopened until at sampling depth

Disadvantages:

- Cannot be used to collect liquids that are incompatible with the weight sinker, line or actual collection bottle
- Laboratory supplied bottle may not fit into sampler, thus requiring additional equipment (constructed of PTFE or stainless steel)
- Some mixing of sample may occur when retrieving the sampler from depth

<u>Weighted Bottle Sampler Use Procedures:</u>

1. Assemble the sampler.
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 evidenced by the cessation of air bubbles.)
4. Retrieve sampler.
5. Transfer sample into laboratory cleaned sample bottles (if applicable, fill VOA vials first).
6. Follow preservation procedures.
7. Transport sample to laboratory after proper **QA/QC** actions (See **Section 7.6**).

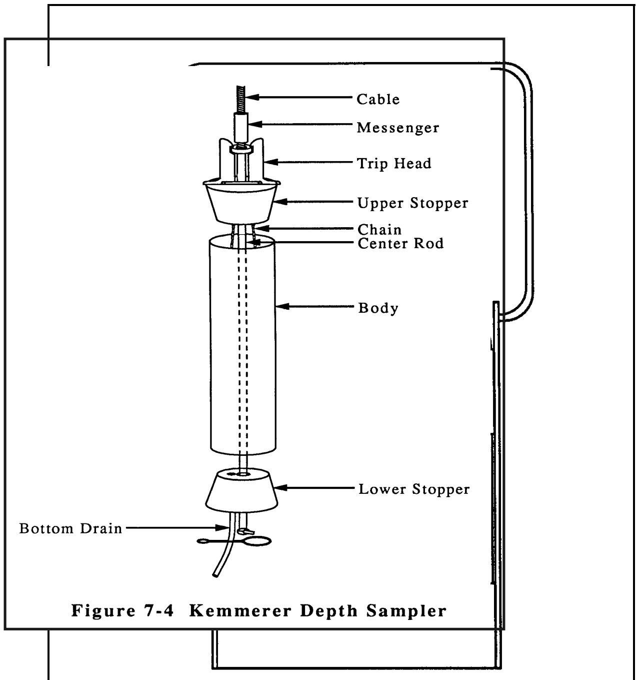

Figure 7-4 Kemmerer Depth Sampler

Figure 7-2 Weighted Bottle Sampler

7.5.1.4 <u>Wheaton Dip Sampler</u>. The Wheaton Dip sampler is useful for collecting samples in shallow areas (See **Figure 7-3**). 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. When the sampler is lowered to the desired sampling depth, the bottle cap is released by turning the metal pole attached to the suction cup. When the bottle is full (usually evidenced by the cessation of air bubbles), the cap is screwed back on to seal the sampling container and the bottle is retrieved.

Advantages:

- Sample bottle is not opened until specified sampling depth is obtained
- Sampler can be closed after drawing sample to ensure sample integrity
- Ease of operation

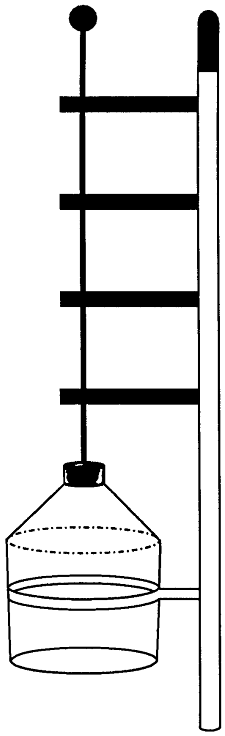

Figure 7-3 Wheaton Dip Sampler

Disadvantages:

- Depth of sampling is limited by length of poles
- Exterior of sample bottle (to be sent to lab) may come in contact with sample

<u>Wheaton Dip Sampler Use Procedures:</u>

1. Assemble the sampler in accordance with the manufacturers' instruction.
2. Operate the sampler several times to ensure proper adjustment, tightness of the cap, etc.

Collect samples for volatile organics analysis first to prevent loss of volatiles due to disturbance of the water.

3. Submerge the sampler into liquid to be sampled.
4. When desired depth is reached, open sample bottle.
5. Once sample is collected, close sample bottle.
6. Retrieve sampler.
7. Follow preservation procedures.

CAUTION

1.

8. Transport sample to laboratory after proper **Quality Assurance/Quality Control (QA/QC)** actions (See **Section 7.6**).

7.5.1.5 Kemmerer Depth Sampler. The Kemmerer depth sampler is used to collect liquid waste samples in lakes, storage tanks, tank trailers, vacuum trucks, or elsewhere, where collection depth prevents use of other sampling devices (See **Figure 7-4**). It 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. These remain in this position until the sampler is at the required sampling depth and then a weighted messenger is sent down the line or cable, releasing the end pieces and trapping the sample within the tube.

Advantages:
- Ability to sample at various depths
- Ability to sample at great depths

Disadvantages:
- Sampling tube is exposed to material while traveling down to sampling depth

If the lagoon or 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 (use of a flat-bottomed boat for increased stability, life preservers, back-up team, etc.) must be implemented.

Kemmerer Depth Sampler Use Procedures:
1. Set the sampler so that the sealing end pieces are pulled away from the sampling tube, allowing the substance to pass through the tube.
2. Lower the pre-set sampling device to the predetermined depth.
3. When the sample is at the required depth, send down the messenger, closing the sampling device.
4. Retrieve sampler.
5. Pour sample into laboratory cleaned sample bottles (if applicable, fill VOA vials first).
6. Follow preservation procedures.
7. Transport sample to laboratory after proper **QA/QC** actions (See **Section 7.6**).

7.5.1.6 Bacon Bomb Sampler. The Bacon bomb sampler is a widely used, commercially available sampler designed for sampling petroleum products (See **Figure 7-5**). It is very useful for sampling large storage tanks because the internal collection chamber is not exposed to product until the sampler is triggered. 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. These range in volume from 4 oz. to 32 oz. It is equipped with a spring loaded trigger. When opened, the trigger allows liquid to enter the collection chamber. When the trigger is released, liquid is prevented from flowing into or out of the collection chamber.

WARNING

Advantages:
- Sampler remains unopened until at sampling depth

Disadvantages:
- Difficult to decontaminate
- Difficulties in transferring sample to container
- Tends to aerate sample
- Brass construction may not be appropriate in metals analysis or toxicity testing

Bacon Bomb Sampler Use Procedures:

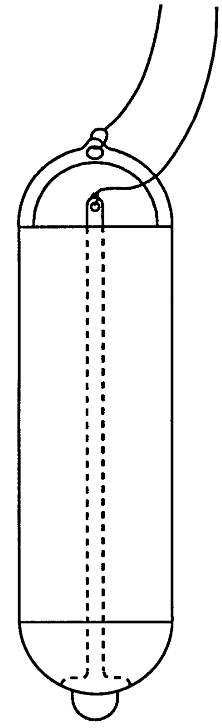

Figure 7-5 Bacon Bomb Sampler

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.
2. Release the trigger line and retrieve the sampler. Pour the sample into the laboratory cleaned sample container by pulling upon the trigger. If applicable, fill the VOA vials first.
3. Follow preservation procedures.
4. Transport sample to laboratory after proper **QA/QC** actions (See **Section 7.6**).

7.5.1.7 <u>PACS Grab Sampler</u>. The PACS Grab sampler can be used to collect water and liquid waste samples from lagoons, ponds, or containers with restricted access

(See **Figure 7-6**). For water and liquid waste

sampling, the narrow neck model is useful. The sampler consists of a 1000 mL bottle screwed onto the end of a six foot long handle. The control valve is operated from the top of the handle once the sampler is at desired depth.

Advantages:

- Allows discrete samples to be taken at depth.

Disadvantages:

- Depth of sampling is limited by length of pole.
- Difficult to decontaminate.

<u>PACS Grab Sampler Use Procedures:</u>

1. Assemble the sampler in accordance with the manufacturers' instruction.
2. Operate the sampler several times to ensure proper adjustment, tightness of the cap, etc.
3. Submerge sampler into liquid to be sampled.
4. When desired depth is reached, open sampler bottle.
5. Once sample is collected, close sample bottle.
6. Retrieve sampler.
7. Pour sample into laboratory cleaned sample bottles (if applicable, fill VOA vials first).
8. Follow preservation procedures.
1. Transport sample to the laboratory after proper QA/QC actions (See **Section 7.6**).

7.5.2 <u>Collection Procedures</u>.

7.5.2.1 <u>Sampling for Volatile Organic Chemicals (VOCs)</u>.

1. Remove the cap from a 40-mL septum (Teflon®-faced silicon rubber) vial. Avoid contact with the inner surface.
2. When acid preservation of VOA is required follow steps 2 to 4. An extra VOA vial should be used to determine the minimum amount of Hydrochloric acid (HCl) required to bring the sample pH to < 2.
3. Fill the vial with sample water, then add 1:1 HCl drop by drop to VOA vial and test pH until it is < 2. Record the amount of HCl added.

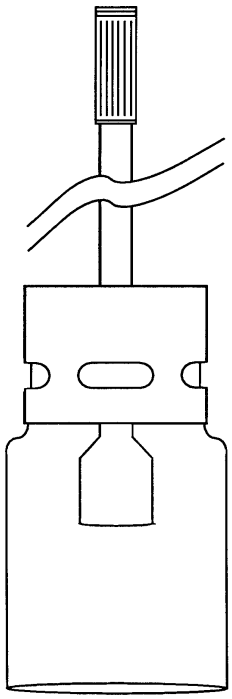

Figure 7-6 PACS Grab Sampler

4. Add the established amount of HCl to each remaining VOA vial and screw vial tightly to achieve zero headspace.
5. Inspect the VOA vial for air bubbles. If air bubbles are present then discard the vial and start again at step 1.
6. Attach a number label and tag to the vial, seal it in a resealable bag and place it into a cooler with ice. Place sufficient ice bags in the cooler to completely surround the samples and to maintain a temperature of 4°C until the samples are received by the laboratory.
7. Record all appropriate data in Field Log Book/Field Notes.

7.5.2.2 Samples for Extractable Organic Chemicals.

1. Remove the Teflon®-lined cap from a one liter, amber, glass bottle. Avoid contact with the inner surface of the cap.
2. Fill about 80% of the bottle with surface water. Add chemical preservatives as required by the permit or method. Record the amount, preservative and other preservation data per the FSP.
3. Replace the cap tightly, attach the sample label, and place the sample bottle in a cooler with bagged ice sufficient to cool to 4°C.
4. Fill the additional bottles by repeating steps 1 through 3.
5. Record all appropriate data in a Field Log Book/Field Notes.

7.5.2.3 Sampling for Metals.

1. Fill the bottles for metals analysis to about 90% and preserve to a pH < 2 with Nitric Acid.
2. Record the amount, preservative and other preservation data per the FSP. Replace the cap tightly, attach label, and place the sample bottle in a cooler with bagged ice sufficient to cool to 4°C, if required.
3. Fill any additional bottles required for separate processing about 90%. Preserve as required by the FSP. Repeat step 2 above. (See **Appendix H** for guidance on various preservation methods.)
4. Record all appropriate data in Field Log Book/Field Notes.
5. Pack samples as noted in **Section 7.5.2.1** step 6 and 7 for sample collection and ship.

7.5.2.4 Sampling for Other/Additional Parameters.

1. Remove caps from the bottles.
2. Fill the bottles per the FSP or method requirements.
3. Add appropriate preservative to the samples (see **Appendix H**).
4. Replace the caps tightly.
5. Attach label.
6. Place sample bottles in a cooler with enough bagged ice to cool them to 4°C, if necessary.
7. Record all appropriate data in the Field Log Book/Field Notes.

7.5.2.5 Autosamplers. Follow the manufacturers recommendations for proper calibration and operation of equipment prior to sample collection.

7.6 QUALITY ASSURANCE/QUALITY CONTROL. 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 method desired in a potable or nonpotable water matrix. All samples must be accompanied by a complete **Chain-of-Custody (COC) Record**. The Field Blanks, Trip Blanks, Field Decontamination Blanks and Field Duplicates should be collected as part of a QA plan to enable data evaluation for accuracy and integrity of surface water sampling. The field decontamination process must be followed properly to ensure QC of the field sampling.

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

Main elements of the Quality Assurance Plan (QAP) for surface water sampling are:

- QA objective for measurements
- Sampling procedures
- Sample custody
- Calibration procedures, if necessary
- Analytical procedures, if necessary

- Data reduction
- Internal quality control
- Performance audit
- Data assessment or validation, if necessary
- Quality assurance report
- Corrective actions

Two Field Duplicate samples and a field equipment blank sample, if necessary should be collected and analyzed. Matrix spikes and matrix spike duplicates may be performed one time per matrix to validate the method selected with the matrix being analyzed. More or less frequent QC may be included in the QAP of the site-specific FSP.

7.7 <u>SAMPLE EQUIPMENT LIST</u>. **Chapter 4, Section 4.8** provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to surface water sampling:

☐ Preservation chemicals and reagents
☐ Materials for decontamination and field blanks
☐ pH meter, dissolved oxygen (DO) meter, conductivity meter, and thermometer
☐ Pond Sampler
☐ Weighted Bottle Sampler
☐ Wheaton Dip Sampler
☐ Kemmerer Depth Sampler
☐ Bacon Bomb Sampler
☐ PACS Grab Sampler
☐ Stainless steel tape and measuring rod
☐ Trip Blanks when measuring volatile organics
☐ Cleaning materials and reagents
☐ Decontamination detergent
☐ Dedicated, pre-cleaned stainless steel pitchers, or equivalent dipping devices, if necessary
☐ Camera, if necessary
☐ Personal Protective Equipment

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 8

GROUNDWATER SAMPLING

8.1 PURPOSE. This chapter provides procedures for obtaining representative samples of groundwater.

8.2 SCOPE. These procedures describe recommended methods as well as minimally acceptable methods for obtaining representative groundwater samples for organic, inorganic, residue, nutrient, bacteriological, and other general chemical analysis. Groundwater monitoring wells, homeowners' private supply wells, underground injection wells, industrial or municipal supply wells are the potential sources of these samples. This chapter includes the minimum criteria to be followed to obtain representative samples. Variations from this criteria should only be necessary when required by regulatory practices or site historical data gathering practices. Analytical data derived from samples obtained in a way which does not follow the documented sampling plan should not be accepted. For construction and design of groundwater monitoring wells (See **Figure 8-1**), reference should be made to **ASTM D5092-90, *Well Design and Construction*.**

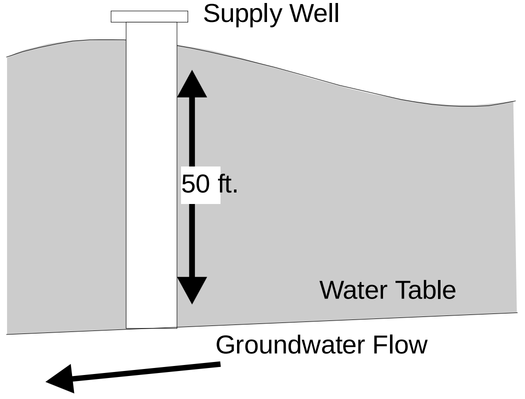

Figure 8-1 Supply Well

8.3 HAZARDS AND SAFETY PRECAUTIONS. See **Chapter 3, Section 3.3.3**.

8.4 PREPARATION. The equipment utilized for specific groundwater sampling events can vary greatly, depending on the following factors:

- Type of well (e.g., monitoring well, supply 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

A relatively new development in groundwater sampling technology has been the design of **in situ** sampling probes, which allow collection of groundwater samples without the installation of permanent wells. A hydropunch operates in conjunction with conventional cone penetrometer rigs. This category 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 deciding the best location of permanent monitoring wells.

Equipment to be utilized for groundwater sampling generally fall into two categories: (a) that used to evacuate water in the well casing, and (b) that 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.

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 which minimizes agitation, air content, gas exchange, and depressurization is preferred.

Sampling devices must be cleaned, preferably by the laboratory performing the analysis, utilizing recommen-ded equipment cleaning procedures. The sampling device should be dedicated to the individual well location for one day of sampling and wrapped in appropriately cleaned aluminum foil or paper. The sampling device should remain wrapped in this manner and stored in an area where contamination will not occur prior to its use. Down hole devices 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.

Types of equipment available for well evacuation include:

- 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

In addition to evacuation and sampling devices, other equipment necessary for a sampling event is included at the end of the chapter.

8.5 SAMPLING PROCEDURES.

8.5.1 Sampling Monitor Wells. Evacuation of the water column in a monitor well is required prior to sample collection. This removes the standing water column and induces 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 phase non-aqueous phase liquids or stagnant water.

Access to monitor wells may be difficult and the wells themselves hard to locate in the field. Obtain information on the location, access, permission, etc. before visiting the site. Monitor wells usually have a friction cap or screw cap, and should be locked. Therefore, keys to unlock the wells and tools for removing caps are often necessary.

If several monitor 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 is essential to avoid confusion of sample results. When several monitor wells of known or suspected contamination will be sampled, the least-contaminated well should be sampled first, and thereafter, sampled in ascending order of contamination. Well head readings using photo or flame ionization detectors (PIDs or FIDs) can aid in determining the order in which wells should be sampled by providing information on levels of contamination.

8.5.2 Field Measurements.

8.5.2.1 Water Level Measurements. Well depths and water table depths can be determined by various measuring devices. A commonly used device is the electronic water level indicator. These units have a tape divided into incremental measurements of 0.01 feet, and two conductors forming a probe. When groundwater is encountered, the circuit is complete 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 monitor well.

Water indicator paste/gel acts as a colorimetric test method when the paste comes in 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 one 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 good 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 from the air to the hydrocarbon surface to prevent 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 would also enhance the hydrocarbon thickness reading. This is best done by lowering the probe 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. Readings should be made three to four times. All well measurements should be performed the same day and prior to evacuation of any wells which may 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. Alternative solvents may be used upon approval of the customer.

Once a well has been located and properly identified, the field measurements listed below should be noted in the **Field Log Book/Field Notes**.

CAUTION

Be certain that the proper well is being selected.The misidentification of a sampling point in the field will result in false data that may affect important decisions.

8.5.2.2 Physical Measurements.

- 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).
- 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 sampling the first well, thus avoiding interference problems. This also allows one to determine if any well is damaged or may pose a problem for sampling.

The capacity of common casing diameters are as follows:

The amount of water within the well casing is calculated by multiplying the linear feet of water by the volume per foot for the proper diameter casing.

Casing Diameter (ft.)	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.

The amount of standing water in the casing should then be multiplied by three (3) to determine the minimum volume to be purged from the well prior to sample collection. The total volume purged should not exceed five (5) times the amount of standing water in the well.

Alternately, one can use this formula to determine the gallons in any diameter well:

Number of gallons = 5.8752 x C^2 x H
where: C = casing diameter in feet and
H = height of water column in feet.

8.5.2.3 Physio-Chemical Parameters. In addition to the physical measurements taken above and other information that may identify the well, information including specific conductance, pH, temperature, and turbidity may be recorded during well evacuation and before and after sample collection.

8.5.3 Well Purging or Evacuation Procedures.

8.5.3.1 Theory. Obtaining representative groundwater samples from monitor wells is required for groundwater pollution investigations. The length of time (stabilization period) for groundwater conditions to become representative at and near the monitor well will vary, depending on site hydro-geologic conditions, drilling methods, and monitor well development methods. Groundwater flow velocities are typically less than one foot per day and natural flushing rates are generally slow. If a monitor 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 one foot per day, it would take 14 days for representative groundwater to reach the well. Sampling a monitor well immediately after development will generally not be representative of the static groundwater quality conditions at the horizontal and vertical location of the monitor well's intake interval. Therefore, all newly constructed and developed monitor wells must be allowed to stabilize and equalize with the aquifer for a minimum of two weeks prior to sampling.

Monitor well development is required to:

- Remove drilling fluid residues remaining in the bore hole or surrounding aquifer.
- Remove imported drilling water lost to the aquifer during the drilling procedure.
- Remove groundwater in the bore hole or surrounding aquifer which has been affected by the drilling process or drilling or well construction materials.
- Restore the hydraulic properties of the formation immediately surrounding the monitor well.
- Allow groundwater to freely flow to the monitor well.

Installation and construction of monitor wells may, by themselves, 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 it is a combination of the techniques mentioned below which will be necessary to produce a properly developed monitor well. Also discussed are certain outcomes inherent to well development techniques which 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 monitor well. This air has the potential for altering groundwater quality, particularly for VOCs.
- Over-pumping of a monitor well for development may draw groundwater to the monitor well from considerable distances. This water may not be of the quality representative of the horizontal and vertical location required by the monitor 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 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 monitor well location during development, depending on the development method, resulting in non-representative groundwater samples being obtained.
- Suspended sediment in groundwater of a monitor well which is 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 monitor 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 monitor well's design, construction, and hydro-geological setting is necessary in order to properly interpret any analytical results.

The well evacuation procedure allows representative groundwater to enter the well. Air sensitive parameters such as dissolved oxygen, pH, temperature, and specific conductance are best analyzed with the use of a flow-through cell, eliminating sample exposure and influence by air. However, monitoring of these air sensitive parameters for well stability may not be a reliable indicator as to when to collect a representative sample. Therefore, if a constant monitor is not used during well purging, a sample should be collected within two hours after three to five 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 must be made to keep pumping rates low to avoid over-pumping or pumping the well to dryness. To accomplish this, pumped rates may be adjusted and pumping times extended in order to remove the required three to five well volumes. In no case should the time of sampling exceed 24 hours after purging. To avoid altering the hydro-geological properties of the aquifer in the vicinity of the well, the evacuation rate of a monitor well should not exceed that of the development of the well.

In some situations evacuation of three to five volumes may be impractical in wells with slow recoveries. If a well has been pumped to near dryness at a rate 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 two 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 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 volatile organic chemicals (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 due to one or more of the above effects.

There are certain circumstances where a well should not be screened across the water table, such as the following:

- Wells screened for collection of depth discrete groundwater samples.
- Bedrock wells with several water-bearing zones.
- Very slow recovering wells.

In these circumstances, the well must not be pumped as to allow the groundwater level to fall below the zone where water enters the well. If a well is evacuated to dryness or below the well screen, sample records should document the event since sample integrity may be severely altered.

8.5.3.2 **Evacuation Methods.** Many methods may be used for well evacuation. 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 less than 25 feet) and a submersible pump (when the depth to water is greater than 25 feet).

It is important to ensure that the evacuation procedure 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. Since in many cases it may not be practical to dedicate a pump to a specific well, it is permissible to decontaminate this equipment between wells, if approved methods are used. Tubing should always be dedicated to each individual well.

Prior to evacuation, check the well for floating product. During evacuation, the pump intake or tubing should be kept at a maximum distance of six feet below the water level. The pump intake or tubing should be lowered as the water level decreases to maintain this distance. In instances where the total depth of standing water in the well casing is less than six feet, begin evacuation near the top of the water column and lower as stated above. Following this procedure 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.

CAUTION

Regardless of the evacuation procedure used, the evacuation rate should not exceed that of well development. This 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 (e.g., gasoline pumps). If this should occur, the item should not be placed in the well or utilized for evacuation.

Finally, the following information should be recorded in the **Field Log Book/Field Notes** 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
- Check for free product, measure thickness if present
- pH, dissolved oxygen, 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, dissolved oxygen, temperature, and specific conductivity

After Sampling:

- Start and end time for sampling
- pH, dissolved oxygen, temperature, and specific conductivity
- Sampling method

Any comments concerning field observations during the groundwater sampling event (e.g., slow recharge, turbidity, odor, sheens, PID or FID readings, etc.) should also be reported.

8.5.3.3 <u>Evacuation Procedure Using Suction Lift Pumps/Centrifugal Pumps</u>. Suction lift pumps (i.e., diaphragm and centrifugal) are pumps utilized at the ground surface with polyethylene tubing inserted into the well. They are used to evacuate the well prior to sampling. The tubing must be new and dedicated to a particular monitor well. The tubing should be equipped with a decontaminated foot check valve to avoid having aerated water from the pump fall back into the well.

If a foot check valve is not used, care must be taken to ensure that the entire pump impeller chamber is drained after being used and then thoroughly decontaminated. Also, when removing tubing without a foot check valve after evacuation, the pump must continue to operate to keep purged water remaining in the tubing and pump chamber from falling 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.

CAUTION

Care must be taken as to the source of the water used in priming the centrifugal pump; ONLY ASTM Type II or better quality water free of chlorine residual and potential contaminants should be used.

NOTE:

These pumps may only be used for well evacuation, **not** for groundwater sampling.

8.5.3.4 <u>Evacuation Procedure Using Portable Submersible Pumps</u>. 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 utilized. The pump must be carefully lowered 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 most of the weight of the pump. These items can be bundled together at ten 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, lower the submersible pump to the appropriate depth. The pump can then 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 not be operating while a sample is being collected.

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 fall 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 from the previous well occurs.

CAUTION

Submersible pumps are susceptible to clogging. Turbid groundwater or poorly developed monitor wells are likely to impede the evacuation process. Care should be taken not to let the pump draw from the bottom of the well where silts and sands may be taken up by the pump.

8.5.3.5 <u>Evacuation Procedures Using Peristaltic Pumps</u>. A peristaltic pump is a self priming suction lift

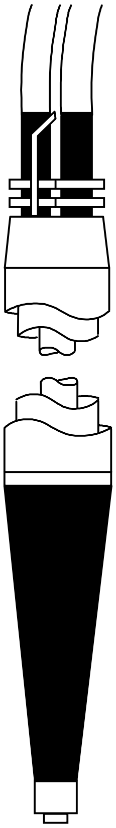

Figure 8-2 Bladder Pump

pump, utilized 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 which has been threaded around the rotor, out of the pump, and connected to a discharge tube. The liquid moves totally within the sample tube, with no part of the pump contacting the liquid. Tubing used for well evacuation may also be used for sample collection. Flexible polytetrafluoroethylene (PTFE) tubing is recommended for sampling. However, other materials may be acceptable, with approval on a case basis. The bottom length of tubing should be equipped with a foot check valve to avoid having water from the pump and tubing fall 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 greater than twenty-five feet. Also, due to the volume present in large diameter and high yield wells, peristaltic pumps are not recommended.

8.5.3.6 <u>Evacuation Procedure Using Air Lift Pumps</u>. This method is generally used for well development and is not recommended for well evacuation prior to sampling. If logistics dictate that air lift pumps are the only alternative to evacuation, then the procedure can only be applied to wells screened below the water table. Tubing connected to air lift pumps must be placed above the well screen since air may become trapped in the screen and/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.

8.5.3.7 <u>Evacuation Procedures Using Bladder Pumps (Gas Squeeze Pumps)</u>. A bladder pump consists of a stainless steel cylindrical housing that encloses a flexible membrane (See **Figure 8-2**). Below the bladder, a screen is attached to filter any material that may clog the check valves that are located above and below the bladder. The pump works as follows: Water enters the membrane through the lower check valve, 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 goes out 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. There is no contact of compressed gas with the sample water.

The bladder pump is utilized much like a portable submersible pump, except that no electrical lines are lowered down the well. The source of gas for the bladder is either bottled gas or an on-site oil-less air compressor. Disadvantages 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 PTFE (Teflon®) or stainless steel.

8.5.3.8 <u>Evacuation Procedure Using Packer Pumps</u>. 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 for evacuation prior to sampling. Also, several zones within a single well can be sampled.

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.

The use of packer pumps for evacuation must be approved by the customer on a case basis. The sampler must be sure the zone being sampled and packed is isolated from the other zones.

8.5.3.9 **Evacuation Procedures Using 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 entry during the suction stroke and forces the water to the surface during the pressure stroke.

The use of gas piston pumps for evacuation must be specified in the Field Sampling Plan (FSP) on a case basis.

8.5.3.10 **Evacuation Procedures Using Gas Displacement Pumps.** Gas displacement pumps work by gas forcing 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 seats and water is forced out the discharge line. When the air pressure is released, the pump chamber fills and the cycle repeats.

The use of gas displacement pumps for evacuation must be specified in the FSP on a case basis.

8.5.3.11 **Evacuation Procedures Using 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. This causes the water inside the pump to be moved up a distance due to its inertia. This can be accomplished manually or automatically utilizing a powered unit.

The advantages of this type of pump are its ease of operation and inexpensive cost. It has several disadvantages, such as:

- 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 the physical act of purging the well. Conversely, the inertial pump device may be overwhelmed in a rapidly recharging well leading to insufficient evacuation of the water column.

The use of inertial pumps for evacuation must be specified in the FSP on a case by case basis.

8.5.3.12 **Evacuation Procedures Using Hand Bailing Techniques.** Hand bailing may be utilized if no other method of evacuation can accomplish the task and the procedure is specified in the FSP. However, bailing is the **least recommended** procedure for well purging due to the potential to aerate the well water or possibly introduce contaminants during the bailing procedure. Specifically, 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 PTFE or stainless steel bailer. An additional laboratory cleaned and dedicated bailer should be required for sample collection.

NOTE:

Hand bailers come in a variety of sizes and volumes to accommodate most well casing diameters.

The bailer must be slowly lowered into the well, exercising care not to aerate the groundwater to be sampled. The preferred method is by using a Teflon®-coated, stainless steel cable attached to a low-gear-ratio winch which is connected to a tripod standing over the well. This is the most reproducible method of bailing a well. If this apparatus is not available, the bailer may be lowered by hand using a Teflon®-coated, stainless steel leader. (Due to the manufacturing oils associated with braided stainless steel cable, Teflon®-coated, stainless steel is required for the bailer leader contacting groundwater, unless decontaminated single strand stainless steel cable is utilized. Lower the bailer until it is submerged. Retrieve it and transfer the water to a container or other device to measure the volume evacuated. The bailer utilized for well evacuation along with any other equipment entering the well for sample collection must be handled with new surgical gloves to prevent potential contamination. It is good to have extra laboratory-cleaned bailers available at the site.

8.5.4 **Groundwater Sampling Procedures.** After evacuation of the required volume of water from the well, sampling can begin. If the well is a quick recharger, sampling of the well should occur as soon as possible after evacuation, preferably immediately. In most cases, the time lapse between evacuation and sampling should not exceed two hours. When several wells are to be sampled of known or suspected contamination, the least contaminated well should be sampled first, with remaining wells then sampled in ascending order of contamination. Well head readings can aid in determining sample order by providing information on contaminant levels in the wells.

Attention to decontamination procedures must be strictly followed. Information on the various methods of sample collection is provided as follows:

8.5.4.1 Sampling Procedures Using Bottom Fill Bailers. Bailers come in a variety of sizes and volumes to accommodate most well casing diameters (See **Figure 8-3**). The preferred materials of construction are PTFE (Teflon®) and stainless steel. The use of bailers constructed of other materials for groundwater sample collection must be specified in the FSP on a case basis.

The bailer must be cleaned and wrapped using approved methodologies, preferable by the laboratory performing the analysis. The bailer must be slowly lowered into the well, exercising care not to aerate the groundwater to be sampled. The preferable method is by the use of a Teflon®-coated, stainless steel cable attached to a low gear ratio winch which is connected to a tripod standing over the well. If this apparatus is not available, the bailer may be lowered by hand using three to six feet of Teflon®-coated, stainless steel leader wire attached to the bailer and to an appropriate length of dedicated polypropylene rope.

> Due to the manufacturing oils associated with braided stainless steel cable and its decontamination difficulty, Teflon®-coated, stainless steel is required for the bailer leader contacting groundwater.

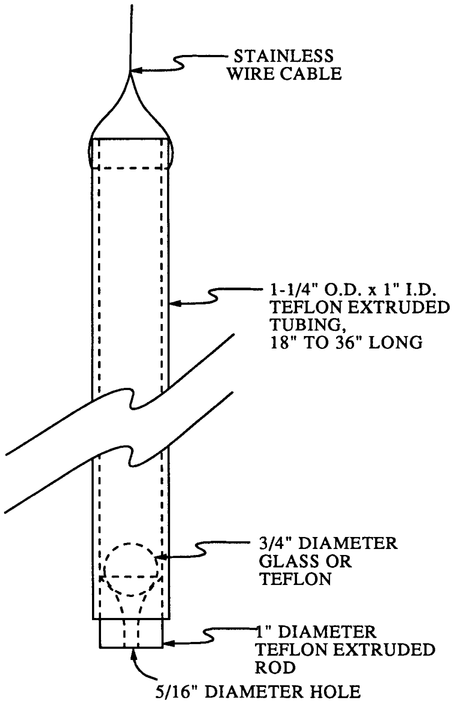

Figure 8-3 Bottom Fill Bailer

Care should be taken if using stainless steel cable clamps when securing the leader to the bailer. The integrity of the Teflon® may be compromised by compression while tightening the clamps, thus exposing the braided wire. Also, all cut ends of leaders must have an end cap so as to eliminate exposure of the stainless wire.

Slowly lower the bailer into the well until it is submerged. Retrieve it and transfer the sample to appropriate containers. Caution must be used in transferring the water from the bailer to the sample container because this action allows the greatest chance of sample aeration.

CAUTION

> Due to manufacturing oils associated with braided stainless steel cable and it's decontamination difficutly, Teflon®-coated stainless steel is reqired for the bailer leader contracting groundwater.

Some bailer manufacturers have small stopcocks with an attached sample line. The valve is inserted into the bottom of the bailer, pushing the check valve up and supplying water to the sample line. The sample flow for the VOCs may then be reduced to eliminate aeration of the sample. The valve should be in the open position when inserting into the bailer, after which it may be closed. This procedure should prevent an air bubble from rising up inside the bailer through the sample, thereby causing aeration.

The order in which samples should be collected from each well, regardless of sampling device, is as follows:

The first bailer recovered after well evacuation must be utilized for sample collection.

1. Volatile organic analytes (VOAs)
2. Purgeable organic carbons (POCs)
3. Purgeable organic halogens (POXs)
4. Total organic halogens (TOXs)
5. Total organic carbon (TOC)
6. Base neutrals/acid extractables
7. Total Petroleum Hydrocarbons (TPH)/ Oil and Grease
8. Polychlorinated biphenyls (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 the Resource Conservation and Recovery Act (RCRA) and the *Groundwater Monitoring Technical Enforcement Guidance Document (TEGD)*, September 1986.

The bailer and any other equipment entering the well must be laboratory-cleaned and handled with new surgical gloves to prevent potential contamination. Surgical gloves must be changed between each sample locations. Clean sampling equipment and any other objects entering the well should not be allowed to contact the ground or any other potentially contaminated surfaces (e.g., gasoline pumps). If this should occur, that item should not be placed in the well or utilized for sampling. It is good to have extra laboratory cleaned bailers available at the site. Additionally, bailers and sample bottles must be physically separated from pumps or generators during transport and storage.

CAUTION

Dedicating a bailer and leaving it in a well for long term monitoring is not recommended due to the potential risk of contamination resulting from excessive handling (it would be necessary to remove bailer first in order to purge the well therefore increasing the risk of contamination).

8.5.4.2 <u>Sampling Procedures Using Peristaltic Pumps</u>. A peristaltic pump is a self priming suction lift pump which consists of a rotor with ball bearing rollers. It is operated at the ground surface. One end of the dedicated tubing is inserted into the well and the other end is attached to a flexible tube which has been threaded around the rotor, out of the pump and connected to a discharge tube. PTFE and polyethylene are the preferred materials for the tubing associated with a peristaltic pump. Other material may be acceptable, particularly for threading through the pump, but must be specified in the FSP on a case basis. The sample moves totally within the sample tube and no part of the pump contacts the liquid. If Teflon® tubing was used for well evacuation, the same length of tubing may be used for sample collection at that well. If another approved material was used for well evacuation, a new dedicated piece of Teflon® tubing may be required for sample collection.

The tubing should be equipped with a foot check valve to avoid having water from the pump and the tubing fall back into the well. A foot check valve is not required if the pump is not shut off in between evacuation and sampling of the well however, a foot valve is still desirable in case of pump failure.

Value of a peristaltic pump for well sampling may be questionable due to its limited suction capabilities. Generally, it cannot be used to sample wells with a depth to water greater than 25 ft.

NOTE:

This pump cannot be used to collect samples for volatile organics or base neutral/acid extractable organics due to the pressure gradients to which the sample is exposed.

8.5.4.3 <u>Sampling Procedures Using Bladder Pumps (Gas Squeeze Pumps)</u>. A bladder pump consists of a stainless steel cylindrical housing that encloses a flexible membrane. Below the bladder, a screen is attached to filter any material which could clog the check valves located above and below the bladder. The pump works as follows: Water enters the membrane through the lower check valve. 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 it closes the lower check valve and goes out 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. There is no contact of compressed gas with the sample water.

The bladder pump is utilized much like the portable submersible pumps, except that no electrical lines are lowered down the well. The source of gas for the bladder is either bottled gas or an on-site oil-less air compressor. The preferred material of construction for bladder pumps and any tube, joint or other fixture that remains in contact with the groundwater, is PTFE or stainless steel.

All pumps and fixtures must be laboratory cleaned prior to installation or use and dedicated to a particular well.

NOTE:

The same bladder pump may be used for well evacuation and sample collection provided that Teflon® tubing is used.

NOTE:

Bladder pumps are acceptable to use for the collection of samples for volatile organics and base neutral/acid extractable analysis. Care must be taken to regulate the flow rate during sample collection to avoid surging caused by cycling within the pump.

Disadvantages:

- Large gas volumes needed, especially for lower depths.
- Potential bladder rupture.

8.5.4.4 Sampling Procedures Using 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. 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.

The use of packer pumps to isolate portions of the well for sampling must be specified in the FSP on a case basis as materials of construction may not be appropriate for certain analysis.

Advantages:

- A smaller volume of water is required for evacuation prior to sampling.
- Several zones within a single well can be sampled because the length of the standing water in the column is reduced.

8.5.4.5 Sampling Procedures Using 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 of the pipe.The pump is operated by raising and lowering the tube over a short distance with rapid strokes. This causes the water inside the pump to be moved up a distance due to its inertia. This can be accomplished manually or automatically utilizing a powered unit.

Advantages:

- Ease of operation
- Inexpensive

Disadvantages

- It is manual operation is labor intensive.
- The pump must be dedicated to a well.

CAUTION

Depending upon the static water level within the well, care must be taken during evacuation and sampling so as not to aerate the water column. Introduction of ambient air may compromise the volatile organic fraction.

The use of inertial pumps for sampling must be specified in the FSP on a case basis.

8.5.4.6 Sampling Procedures Using Syringe Samplers. The sample container is pressurized or evacuated and lowered into the well. Opening the container and or releasing the pressure allows the sample to enter the device. These systems are not widely used or commercially available. Use must be specified in the FSP on a case basis.

8.5.5 Filtering Groundwater Samples. In order to ensure quality of data generated from analysis of groundwater samples, critical sample handling procedures must be addressed. Of chief importance is sample filtration. Because objectives may vary among monitoring programs, it is difficult to establish a filtering standard that applies to all situations.

Regulations **require** metals analysis to be performed on **unfiltered** groundwater samples pursuant to the requirements of the **Safe Drinking Water Act** and the **Clean Water Act**. The reason for this is to obtain a representative sample as it actually occurs in the aquifer and to maintain consistency between sample handling for inorganic and organic analysis. If a particular case demands consideration of dissolved metals, both filtered and non-filtered samples should be collected for analysis. The regulatory document or approved quality assurance project plan 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 whether to filter metal(s) samples should be based on the physical quality of the samples, the objective of the monitoring program, and the policy of the Program controlling the specific 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 consistent reproduction of technique.

8.5.6 Sampling for Volatile Organics.

1. Remove the cap from a 40-mL septum (Teflon®-faced silicon rubber) vial. Avoid contact with the inner surface.
2. Fill the vial with sample water, then add 1:1 HCl drop by drop and test the pH until it is < 2. Record the amount of HCl added.
3. Add the established amount of HCl to each remaining VOA vial and screw cap on tightly to achieve zero headspace.
4. Inspect the VOA vial for air bubbles. If air bubbles are present, then discard the vial and restart with step 1.
5. Attach a number label and tag to the vial, seal it in a resealable bag and place it into a cooler with ice. Place sufficient ice bags in the cooler to completely surround the samples and to maintain a temperature of 4°C until the samples are delivered to the laboratory.
6. Record all appropriate data in Field Log Book/Field Notes.

8.5.7 Sampling for Extractable Organics.

1. Remove the Teflon®-lined cap from a one liter amber glass bottle. Avoid contact with the inner surface of the cap.
2. Fill about 80% of the bottle with ground water.
3. Replace the cap tightly, attach the sample label and place the sample bottle in a cooler with sufficient bagged ice to cool to 4°C.
4. Repeat steps 1 through 3 for additional samples.
5. Record appropriate data in a **Field Log Book/ Field Notes**.

8.5.8 Sampling for Dissolved Metals and Cyanide.

Filtration of groundwater samples for dissolved metals analysis should be performed with a precleaned filtering apparatus. Sampling devices should be cleaned using ultrapure nitric acid when low level contaminants are being measured. Devices such as polyethylene or borosilicate glass should be used when filtering the groundwater samples for inorganic analysis. Filtration must be done immediately upon sample collection, prior to preservation. Samples transported to the lab for filtration and preservation should be documented since sample composition will change during transport. The sample should be collected and filtered with 0.45 micron pore diameter cellulose acetate filter. If the use of a vacuum filter is impractical, pressure filtration must be performed. Care must be taken to strictly follow the manufacture's recommended procedure if vacuum filtration is used. All filter apparatus should be laboratory-cleaned and dedicated. Disposable filters are acceptable. For each sampling event, a new disposable filter must be used to avoid cross-contamination of samples. The following guidelines apply to samples collected for trace metal analysis of groundwater:

- Groundwater samples for metals may be filtered using in-line filtration devices or vacuum filtration in the field. Unfiltered samples should represent "worst case" with respect to metal content. Regulatory or permit requirements will indicate when filtered and unfiltered samples are to be collected.
- If metal concentrations are significantly above groundwater standards, some permits require two samples to be collected from each well: one sample filtered according to the procedures and a second unfiltered sample.

NOTE:

Filtered samples are not allowed by the Safe Drinking Water Act program.

1. Fill the bottles for metals analysis to about 90% and preserve with Nitric Acid to a pH < 2.
2. Replace the cap tightly, attach label, seal in resealable bag and place the bottle in a cooler with bagged ice sufficient to cool to 4°C.
3. Fill about 90% of the cyanide sample bottle. Preserve to pH > 12 with NaOH = sodium hydroxide. Repeat step 2
4. Record all appropriate data in Field Log Book/Field Notes.

8.5.9 Sampling for Conventional Parameters.

1. Remove caps from sample bottles.
2. Fill containers for BOD, TOC, TSS, TDS, COD, alkalinity, and chloride to about 90%. Add appropriate preservative to the samples per **Appendix H** or sampling plan.
3. Replace caps tightly, attach labels, seal in resealable bags and place sample bottles in a cooler with sufficient bagged ice to cool to 4°C.
4. Record all appropriate data in a Field Log Book/Field Notes.

The VOC samples are collected first. Care must be taken to prevent volatilization of the sample when placing it in the VOC vial. All **Chain-of-Custody** (COC) procedures should be followed.

Samples are submitted for analysis of VOC's, semivolatile compounds, pesticides, metals, inorganic compounds, bacteriological species, radiological testing, and other parameters required by the permit or regulatory authority. The pH of some samples must be adjusted to preserve the sample for specific analysis. At the completion of each day's sampling, the containers collected during the day are packed on ice in a cooler and sealed if appropriate.

For detail sampling procedures and preservation of samples refer to **Appendix H** of this manual.

8.5.10 Sampling for Light, Non-Aqueous Phase Liquids (LNAPLs). LNAPLS are generally considered to be low density, immiscible organics, including gasoline, petrochemicals, and other chemicals which have specific gravities less than water. They are likely to be present in aquifers as a separate phase because of 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. Thus, if product (LNAPL) is suspected to be floating on the water table, all shallow wells installed in the area under investigation must be screened **across** the water table.

In a confined aquifer, these chemicals are found along the upper surface of the permeable material and also within the overlying confining layer. When immiscible organics with a specific gravity greater 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 bailer used for sample collection to a particular depth in the well, or to utilize a double check valve bailer.Sampling procedures for LNAPL 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 volatile organics and base-neutral extractables, etc.) and physical parameters (e.g., specific gravity, water solubility, vapor pressure of the liquid, and Henry's Law Constant, etc.).

After the well is initially constructed it should be developed and pumped to remove stagnant water, then it should sit idle for at least two weeks to allow the water level to fully stabilize and the floating layer to stabilize.

Measurement of the thickness of the floating layer may be accomplished by using a water indicator paste/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 may also be accomplished by using an interface probe or 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.

Prior to the purging of groundwater from the well, a sample of the floating layer may be obtained using a bailer which fills from the bottom. Care should be taken to lower the bailer just through the floating layer but not significantly down into the underlying groundwater. Samples should be analyzed to determine the chemical composition of the LNAPL and its physical properties (e.g., specific gravity, water solubility, equilibrium vapor pressure of the liquid and Henry's Law Constant, etc.). After following typical evacuation procedures discussed previously in this section, a sample of formation water may be obtained from the well.

8.5.11 Sampling for Dense, Non-Aqueous Phase Liquids (DNAPLs). DNAPLs include chlorinated solvents and other chemicals which have specific gravities greater than water. They are likely to be present in aquifers as a separate phase because of low solubility in water. DNAPL chemicals tend to migrate downward through the unsaturated zone and the saturated zone due to their high density. If the volume of DNAPL chemical 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 (and LNAPLs) must be performed prior to purging (evacuating) the well. Measurement of the DNAPL may be accomplished by using a water indicator paste/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 in the well. An interface probe may also be used to measure DNAPL in the well. An interface probe may also be used to aid the measurement of DNAPL thickness.

Prior to purging a monitor well, a sample of the DNAPL may be obtained using a dual check valve bailer or a bladder pump. If both LNAPLs and DNAPLs are present in a well it may be necessary to purge the well of one casing volume of water prior to sampling the DNAPL, provided that efforts are made not to disturb the DNAPL in the bottom of the well. This can be accomplished 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, etc.). After the well is purged, a sample of the groundwater may be obtained for laboratory analysis.

8.5.12 Sampling Domestic Wells. An important step in sampling a domestic well is to obtain as much information as possible from the homeowner. This should include: 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, determine the number of gallons to be evacuated.

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 fifteen minutes before collection is a good rule of thumb, however, longer is desirable. Listen for the pump or the electric circuit to the pump to come on, indicating that the plumbing is being evacuated. Inquire as to whether any treatment units are installed on the system. Softening, iron removal, turbidity removal, disinfection, pH adjustment may often provide misleading analyses depending on the parameters of interest. Home carbon filters for the removal of organics are also increasingly popular. Basement and outside faucets may by-pass such treated water (Note: sample cold water faucet).

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 on samples sheets and in the Field Log Book/Field Notes.

Home 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, since the screen tends to aerate the water and some organics may be lost. Also, when sampling for bacteria, do not take a sample from a swivel faucet since the joint may harbor a significant bacterial population.

NOTE:

Homeowners' plumbing systems should not be tampered with in any way, except for removal of the faucet screen with permission of 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. The removal of the screen should be noted. In some areas, when sampling for drinking water bacteria, the screen should not be removed since removing the screen is not an indication of the drinking water from the tap.

8.5.13 Sampling Industrial Wells. 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. This should eliminate treatment interferences, possible changes in quality within the lines, mixing of water from other wells, etc.

Large capacity wells which are “on-line” during the visit can be sampled immediately. Wells which are “off-line,” must be pumped to waste prior to sampling. Fifteen minutes or more is suggested. Access to municipal well systems, well houses, etc. requires the assistance of a water department employee. Prior notification is essential.

8.6 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC). Groundwater monitoring and associated laboratory analytical testing demand that sample integrity be maintained during sample collection. Laboratory analysis, no matter how sophisticated, may only be representative if the sample supplied to the analyst has retained its integrity. There are many areas to which specific attention must be paid. Some relate to well drilling and development procedures, well construction materials, or heavy equipment decon-tamination and are discussed elsewhere in this manual. During on-going groundwater monitoring studies, consistency of sampling is crucial to the interpretation of the data over the year or years. Issues addressed in this section include the decontamination procedure and the construction materials used for sampling equipment.

8.6.1 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 into the well. Field measurement equipment, such as water level indicators, should be cleaned in the following manner:

- Wipe with a paper towel to remove visual debris
- Tap water and laboratory grade glassware detergent wash
- Tap water rinse
- ASTM Type II water rinse

Sampling equipment should be laboratory cleaned using documented cleaning procedures, preferably by the laboratory performing sample analysis. The sampling equipment should then be wrapped in cleaned 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 laboratory cleaning should be field cleaned using documented cleaning procedures.

8.6.2 <u>Composition of Construction Materials for Sampling Equipment</u>. The composition of materials comprising groundwater sampling equipment is critical to the collection of valid monitoring information, particularly, when volatile organic, pH sensitive, or valence reduced chemical constituents are being evaluated. The construction materials which come in contact with the sample are as critical as the composition of the laboratory sample containers. Recommended materials for bailers, pump parts, tubing, other sampling devices, and associated apparatus in decreasing order of preference are: PTFE (Teflon®), stainless steel 316, stainless steel 304, polypropylene, linear polyethylene, Polyvinyl Chloride (PVC), Viton, conventional polyethylene.

The majority of regulatory programs require that the bailers be constructed of PTFE or stainless steel. Additionally, any other devices contacting the water to be sampled should be constructed of PTFE or stainless steel. The reader is cautioned that exceptions to this requirement should be confirmed and approved by the regulatory program having project oversight authority.

Tubing utilized in well evacuation may consist of materials other than PTFE, but may not be utilized for sample collection and it is recommended that it should be dedicated for use in each individual well for that particular sampling event.

8.6.3 <u>Quality Control Samples</u>. In an attempt to identify external variables affecting groundwater sample integrity, a program of quality control blanks should be initiated. For volatile parameters, the quality control blank sample program is a two track approach using both a trip and field blank. The trip blank acts as a check on potential contamination sources in sample container preparation, method blank water including preservative, and sample transport and storage. The field blank serves as a check on the cleanliness of the sampling equipment, potential atmospheric contamination, and the effects of sampling procedures on the analytes of interest. The blank water and same preservation materials used in the samples should be used to assess blank contamination problems. Complete documentation on source 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 used and the evaluation of cleaning techniques used in the field.

Field duplicates or splits are collected in the field by collecting double the number of bottles and sending the samples to the same laboratory (duplicate) or a different laboratory (split). This information will determine field precision (duplicate) or project precision (split).

Field spikes are prepared in a limited number of permit or regulatory requirements. Field spikes determine field accuracy or project accuracy. 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 method performance and contaminant deterioration or degradation during sample handling, transport, and analysis.

8.7 <u>SAMPLE EQUIPMENT LIST</u>. Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to Groundwater Sampling:

Water level indicator
Steel line and chalk
Electric tape (i.e., interface probe, slope indicator, M-scope)

Equipment for well evacuation include:

- ☐ 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

Equipment 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

Additional equipment, Optional

☐ Volatile Organics Detection devices such as HNU Photoionization Detector PID or FID
☐ Sample containers (proper size and composition)
☐ Preservatives, as needed
☐ Ice or ice packs
☐ Field and Trip Blanks, as appropriate
☐ Appropriate personal safety equipment (e.g., disposable gloves)
☐ Appropriate hand tools
☐ Keys to locked wells
☐ Filtering devices
☐ Field measurement instrumentation (temp. specific conductance, pH, DO, turbidity, etc.)
☐ Plastic sheeting, ties and bags
☐ Dedicated, precleaned stainless steel pitchers, or equivalent dipping device, if necessary.
☐ Calculator, wristwatch, and timer
☐ Sample shuttle (cooler)
☐ Indelible marker
☐ Calibrated bucket for purged water measurement
☐ Distilled/deionized water or ASTM Type II water
☐ Laboratory grade glassware detergent or cleaning materials
☐ Paper towels
☐ Empty drums for collection of purged water, if necessary
☐ Stainless steel clamps, if necessary
☐ Camera, if necessary

NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 9

DRINKING WATER SAMPLING

9.1 PURPOSE. To assist sampling personnel in collecting drinking water samples using standard procedures that comply with Federal guidelines.

9.2 SCOPE. This chapter provides procedures for sampling ground water and surface water supplies for monitoring drinking water quality. A comprehensive framework designed to ensure the quality and safety of drinking water supplies to the public has been established under the **Safe Drinking Water Act (SDWA)**, and other Federal, state, and local laws. The Federal program sets minimum standards and regulations for collection, treatment, monitoring, storage, and distribution of drinking water. State regulations must be at least as stringent as Federal regulations and may go beyond the minimum criteria established by the Federal program.

The **SDWA** assigns initial responsibility to the EPA and provides states assumption of responsibilities if certain conditions are met. In this chapter regulators refers to the EPA or to the state when granted primacy by the EPA. It is the public water works, water utility company, and public community water system's responsibility to monitor and sample drinking water under Federal and state regulations.

NOTE:

This chapter provides national guidelines for drinking water sampling. Facilities may have special requirements depending on local law. All sources of regulations and permits and their method references must be checked for the proper specifics of sampling, containers, preservation, holding times, and analysis methods. The specifics will change from region to region, state to state, and permit to permit. **Contact state and local governments for sampling requirements.**

The SDWA establishes three general types of public water systems: **Community Water Systems (CWS)**, **Nontransient Noncommunity Water Systems (NTNCWS),** and **Transient Noncommunity Water Systems (TNCWS)**. CWS and NTNCWS follow similar sampling and testing criteria based on the population size or number of service connections within the water supply. In most areas of the country, TNCWS only monitor for bacteria, nitrate, and nitrite.

The drinking water sampling plan should include procedural guidance to perform:

- microbiological, inorganic, and organic sampling
- monitoring of lead/copper at the tap
- monitoring turbidity levels for surface water supplies

The drinking water sampling plan should include a description of the entry point, treatment process, and distribution system. Samples collected from the source water, entry point after treatment, and distribution system may be compared to determine the effect of treatment and the plumbing system on drinking water quality.

A Field Sampling Plan (FSP) should be prepared and reviewed by appropriate regulatory personnel prior to starting any sampling to ensure proper location, parameters, and number of samples are taken for meeting compliance requirements. The specific monitoring and reporting requirements of the SDWA are beyond the scope of this manual.

9.3 HAZARDS AND SAFETY PRECAUTIONS. See **Chapter 3, Section 3.3.3**.

9.4 PREPARATION. Every state certification regulation requires that drinking water samples collected for compliance with the **SDWA** be collected by "approved individuals". Some states require the sampler 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 certified by the local, state, or Federal program with jurisdiction, are allowed to perform compliance testing for microbiology, inorganic, organic, and radiochemistry chemicals. Turbidity, chlorine residual, and pH monitoring are exceptions to the laboratory certification requirements for performing analysis. These tests can be performed by any person acceptable to the state.

The EPA or state certified laboratories should supply the blanks, containers, and preservatives for sampling. The containers, blanks, and preservatives used 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.

NOTE:

Refer to **Section 9.6** and **Appendix I** for quality control sample requirements.

9.4.1 <u>Number and Frequency of Drinking Water Samples</u>. The number of drinking water samples to be collected is dependent upon the water source, the population served, contaminants found during initial monitoring, and source vulnerability. The frequency of repeat sampling is based on detection, vulnerability, source, and system size. The exact number of samples and frequency of sampling is determined in conjunction with local or state regulatory authorities.

The EPA established a nine-year "compliance cycle" for all drinking water parameters. The first cycle began in January 1993. The cycle contains three "three-year compliance periods". This cycle standardizes sampling frequency and waiver deadlines. A complete review of the Federal and state regulatory requirements is required to determine the number, parameters, and frequency of sample collection.

NOTE:

The Federal drinking water regulations can be found in section 40 of **The Code of Federal Regulations** (CFR), part 141 and part 143. Local and state regulations can be obtained from their respective agencies.

9.4.2 <u>Sampling Locations</u>. The location for sample collection depends on:

- the water source
- analyses to be performed
- purpose of the testing
- regulatory requirements

Samples may be collected from the source prior to treatment, at the point of entry (before or after treatment), at the point of use (at the tap), or within the distribution system. For example, VOC sampling is performed at the entry point of the distribution system. Lead and copper samples are taken at the point of use. The appropriate sampling point needs to be determined based on the criteria listed above.

If the water source is ground water, the well is considered the source water. In some cases where a large number of wells are combined into a holding tank, the water from the holding tank is defined as the source water. A review of the water system with the state sanitary engineer or drinking water compliance officer will assist in defining the water source, entry point after treatment, and location within the distribution system for sampling.

9.4.3 <u>Analytical Methods</u>. Methods approved by EPA must be used for samples taken for compliance with regulations. Approved methods are listed in 40 CFR 141 and are also listed in **Appendix H** for your convenience.

9.5 <u>SAMPLING PROCEDURES</u>. The sampling procedures for regulated and unregulated contaminants are presented by group for the most frequently collected parameters. Sampling procedures for new regulated contaminants and other parameters of concern may be found in EPA approved methods, ASTM Water Methods, **<u>Standard Methods for the Examination of Water and Wastewater</u>**, or other published standards. The parameters and method specific quality control (QC) are found in the approved drinking water methods (See **Appendix H** and **I**).

9.5.1 <u>pH, Temperature, and Chlorine Residual</u>. pH, temperature, and chlorine residual measurements are conducted in the field at the start of sample collection. The field testing procedures for pH, temperature and chlorine measurements are presented in **Chapter 14**. Chlorine residual must always 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.

The Free Chlorine Residual method uses the reagent, Diethylphenylenediamine (DPD), in a colorimetric test to determine the amount of free chlorine available for disinfection. Chlorine residual test kits are available for drinking water testing to measure free chlorine and/or total chlorine. 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. Samples must be analyzed immediately, which generally means within 15 minutes of sample collection.

The test kit or method details the exact method for using the kit. An example of a chlorine residual method is found in **Chapter 14**. Most kits use either DPD powder, tablet, or drops of liquid placed in a glass tube containing the sample. A color change after adding the reagent indicates 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. A free residual chlorine of 0.1 ppm is detected by this test 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 may 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 field record (Field Log Book or Field Notes), the results in mg/L, date, time, person, and observations if any. 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.

9.5.2 Total Coliform. The **Total Coliform Rule (TCR)**, which became effective December 31, 1990, supersedes the old **National Interim Primary Drinking Water Regulations (NIPDWR)** for maximum microbiological contaminant levels (effective June 24, 1977). The TCR differs from the old rule in that it is based on the **presence or absence (P/A)** of coliforms rather than the **number** of coliforms detected in the samples. The new rule requires that coliform **positive** samples be further tested for **fecal coliform** or **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.

Each system must have a written sampling plan that lists the frequencies and locations of samples to be collected including repeat samples. Samples for the TCR are collected from the distribution system. This plan may be reviewed and revised by the regulatory agency.

Maximum Contaminant Level (MCL). For systems collecting less than 40 samples per month, no more than one (1) 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.

Number of Samples. The number of samples that must be taken monthly is based on the population served by the water system. **Table 9-1** provides an abbreviated list of **minimum** monthly monitoring requirements. Many community systems routinely sample more than the minimum to keep track of the system's status.

Table 9-1 Monitoring Frequency for Routine Sampling Public Water Systems

Population Served	Minimum Routine Samples/Month
25-1,000*	1
1,001-2,500	2
2,501-3,300	3

NOTE:

CWS serving more than 3300 must refer to the CFR. Regulator may specify a sampling frequency of less than once per month for selected systems. Contact your state drinking water representative for details.

For each routine sample that is total coliform-positive, a system must collect a **set** of repeat samples and have it analyzed for total coliforms. If total coliforms are detected in any routine or repeat sample, the sampler must collect at lease **five** routine samples the next month. **Table 9-2** provides an abbreviated list of the required frequency for repeat sampling.

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

Table 9-2 Monitoring and Repeat Sampling Frequency After a Total Coliform-Positive Routine Sample

Number of Routine Samples Per Month	Number of Repeat Samples	Number of Routine Samples Following Month
1/month*	4	5/month
2/month	3	5/month
3/month	3	5/month

* Or fewer.

If an additional repeat sample is required, it should be taken within five service connections, *either* upstream or downstream of the original sample. If a system has only one service connection, it can collect:

- One 100-mL sample on each of 4 separate days
- Two 200-mL samples on 2 separate days
- One 400-mL sample on 1 day

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. Regardless of analytical method, the analysis must use a minimum 100-mL sample volume per test.

Sampling Containers. Sample containers for collecting coliform samples must be sterilized and at least 120 mL (4 oz), (and contain sodium thiosulfate for chlorinated waters). Bottles may 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 is for protection from contamination. Some labs furnish a single-service, sterilized, polyethylene bag or bottle containing a sodium thiosulfate tablet. The laboratory should perform sterility checks (1 per lot) prior to releasing the containers for collecting samples.

Sampling Procedures. The following instructions illustrate the **general** sampling procedures for collecting coliform analysis monitoring samples. The lab supplying sampling containers normally provides instructions with the bottles for the type of monitoring intended. Refer to those when provided.

1. Assemble all of the sampling supplies before you begin. The proper preservative is added by the laboratory. 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 sampling plan. These representative sampling locations are usually located in the distribution system and are accessible during the day. Examples include hospitals, city buildings, pump stations, restaurants, and dedicated sampling stations. The tap should be clean, free of attachments (hoses, etc.), and in good repair (no leaks). Avoid drinking fountains and faucets with swivel necks unless specific point of use concerns arise.
3. If possible, remove any aerator, strainer, or hose present, as these may harbor bacteria. (You may not be able to remove the aerator or find a non-swivel faucet.)
4. Open the **cold** water tap for about two to three minutes before collecting the sample to clear the service line. (You may want to time this step — three minutes is a long time.)
5. Fill out the label, tag, and lab form in waterproof ink.
6. Adjust the flow to about 500 mL (one pint or two cups) per minute (approximately a 1/8-inch diameter flow). Check for steady flow. Do not change the water flow once sampling has started. It could dislodge microbial growth.
7. For chlorinated systems, check the chlorine residual (refer to **Section 9.5.1**) and record.
8. Remove the bottle cap (stopper, etc.) or open the plastic bag. Be careful not to touch the inside of the container with your fingers. Do not put the cap of the container down on any surface. Position the bottle or bag under the water flow. Hold the bottle in one hand and the cap in the other.

CAUTION

Do not lay the cap down or put it in a pocket! Do not contaminate the sterile bottle (or bag) or 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!**

9. Fill the bottle to the shoulder or to about 1/4 inch from the top ensure that at least 100 mL of sample are collected. If using a plastic bag sampling container, fill it to the marked fill line.

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. Generally samples should be iced immediately although this is not a requirement per 40 CFR 141.21, June 29, 1995.
11. Turn the tap off. Replace the aerator, strainer, or hose.
12. Check that the information on the label is correct.
13. Complete any additional forms that came with the sample bottle, including the **Chain-of-Custody (COC)** Record, with the necessary information. If the laboratory is nearby, ice and deliver the samples there directly. If not, send the samples overnight by U.S. mail or by an overnight courier. All samples shall be refrigerated or iced (cooled to below 10° C). The time from sample collection to initiation of analysis may not exceed 30 hours for distribution sampling and 8 hours for surface water source sampling.

9.5.3 Lead and Copper. The EPA has determined that lead and copper 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. 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 action level (AL). The action level for lead is 0.015 mg/L and for copper is 1.3 mg/L measured at the 90th percentile at the consumer tap. This means that some of your 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.

Location and Frequency. A material survey must be conducted to determine sampling sites. Sampling sites are rated on a tier system. See 40 CFR 141.86(a)3 for specific information on selecting sampling sites.

All systems must be monitored during at least two consecutive six month periods until either:

- They exceed the lead and copper action levels. 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
- They meet the lead and copper action levels for two consecutive periods. Then, the water supply petitions the regulator to reduce the number of tap water sampling sites and reduce the frequency of collection

Number of Samples. The number of samples taken during the six month sampling period is based on the population and results from previous samples. After two six month sampling periods of meeting the action level, a system may request reduced monitoring. (See **Table 9-3**) Monitoring frequency may also be reduced after several years of meeting the action level. Refer to 40 CFR 141 for specific information.

Sampling Containers. Sampling containers for lead and copper may be made of plastic or glass and must be 1-liter in volume. A one-liter plastic bottle is most commonly used.

Table 9-3 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)
3,301 to 10,000	40	20
501 to 3,300	20	10
101 to 500	10	5
up to 100	5	5

Sampling Procedure. There are two types of lead and copper sampling procedures:

- First draw sampling
- Service line sampling

First Draw Sampling. First draw samples are taken to show compliance with the action levels. The water in the plumbing must stand motionless in pipes for at least 6 hours. The following is a detailed sampling procedure for lead and copper sampling:

1. All samples for compliance monitoring must be first draw samples, taken from kitchen cold water or bathroom cold water sink tap.
2. Fill out sample label, indicating the date and time of sample collection, location, type of samples, and sampler's name. The site should be sealed or a certification by the homeowner must be signed indicating the length of time the system was not used. Taps within work site building may 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.

3. **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 may release high amounts of lead and copper into the waterline. Secure taps by taping and posting notices.

4. 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.
5. Fill to the 1-liter mark on the bottle. Turn off the water tap. Samples may be shipped to the laboratory and preserved by the laboratory within 14 days after sample collection.
6. If field preservation is performed, add approximately 3 mL of concentrated ultrapure nitric acid to the sample bottle to preserve the sample. Be extremely careful when adding the acid. Check the sample pH with litmus paper. Indicate if the sample pH is less than 2. If the pH is not less than 2, add acid in 1 mL increments until the pH is less than 2. Record the amount of acid, pH, date, time, person, and acid source. Indicate on the label whether the sample has been acidified.

WARNING

Concentrated acids and other chemical preservatives represent a safety concern. Refer to safety plan or HASP, as appropriate to scope of project, and Chapter 3, Section 3.3.3 for precautions and personnel protective equipment (PPE).

6. Tightly cap the bottle to prevent leakage. For samples collected in a collapsible container, a special insert can be used to provide a better seal.
7. Complete the necessary forms with the appropriate information such as Public Water System (PWS) identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, in distribution system). Also mention the type of analysis to be run. Complete a **COC** Record.
8. Pack the bottles and/or collapsible containers for shipment and return them to the lab for analysis. Samples preserved at the laboratory are not to be analyzed until 16 hours after preservation.

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.

I. If a service line sample is required, there are three ways to help ensure that service line water is being sampled:
    a) At the tap, flush a known volume of water from household plumbing before collecting the sample. This is the water contained in the pipes between the sampling tap and the service lines. Use the container such as a pitcher or bucket for measurement of the volume of water wasted. **Table 9-4** provides an abbreviated list of the volume to be wasted.
    b) For single family homes, allow the water to run until there is a significant change in its temperature. This indicates that you are now getting the water 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 9-4 Approximate Volume in Gallons of Flushwater for Various Sizes and Length of Copper Pipe

Nominal Pipe Size (in.)	Length of Pipe (in.)					
	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

2. After step 1a or 1b, immediately collect the sample in a 1-liter plastic bottle or collapsible container while the water is still flowing. For 1c, turn the service line tap on and collect the water immediately. Samples may be shipped to the laboratory and preserved by the laboratory within 14 days after sample collection.
   If field preservation is performed, acidify the sample with approximately 3 mL of concentrated ultrapure nitric acid. Be extremely careful while

adding the nitric acid. Check the sample pH with litmus paper. Indicate if the sample pH is less than 2. If the pH is not less than 2, add acid in 1 mL increments until the pH is less than 2. Record the amount of acid, pH, date, time, person, and acid source. Indicate on the label whether the sample has been acidified.

WARNING

Concentrated acids and other chemical preservatives represent a safety concern. Refer to safety plan or HASP, as appropriate to scope of the job, and Chapter 3, Section 3.3.3 of this manual for precautions and PPE.

2. Tightly recap the bottle to prevent leakage. If using a collapsible container, a special insert can be used to provide a better seal.
3. Complete the necessary forms with the appropriate information such as Public Water System (PWS) identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, in distribution system). Also mention the type of analysis to be run. Complete a **COC** Record.
4. Pack the bottle and/or collapsible containers for shipment and return them to the lab for analysis. Samples preserved at the laboratory are not to be analyzed until 16 hours after preservation.

9.5.4 <u>Nitrates/Nitrites</u>. Drinking water compliance samples for nitrates/nitrites are taken at the entry point to the distribution system after treatment. Surface water supplies may take samples from the distribution system at a point which is representative of each source after treatment. The MCL for nitrate is 10 mg/L and nitrite is 1 mg/L.

Frequency. Nitrate samples are collected every year for ground water sources and quarterly for surface water sources.

Nitrite samples are collected every three years. Increased sampling is required if results are greater than 50% of MCL.

Number of Samples. The system shall take one sample from each entry point or at points in the distribution system that represent each source or treatment plant.

Sampling Containers. Nitrate samples should be collected in a 1-liter glass or plastic bottle and cooled to 4°C with ice. Samples of non-chlorinated water should be preserved with sulfuric acid to pH < 2 and must be analyzed within 14 days. Nitrate for chlorinated supplies must be analyzed within 28 days.

Nitrite samples should be collected in a 1-liter bottle and chilled on ice, but should not be preserved with sulfuric acid. Samples for nitrite must be analyzed within 48 hours of sample collection.

Sampling Procedures. The general procedure for collecting samples from a tap follows:

1. Determine the appropriate 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 all containers and materials carefully to ensure cleanliness.
2. Go to the sampling location(s) specified in the sampling plan. These representative sampling locations are usually located in the distribution system and are accessible during the day. Examples include hospitals, city buildings, pump stations, restaurants, and dedicated sampling stations. The tap should be clean, free of attachments (hoses, etc.), and in good repair (no leaks). 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, one- liter plastic or glass (as appropriate) container should be used to dip the sample and fill the sample bottle(s).
3. When sampling from a tap, open the tap and allow the system to flush until the water temperature has stabilized (usually 10 min.). Remove hoses and other sources of contamination from around the tap prior to sampling.
4. Fill out the label 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 sample submission form.
5. When sampling from a tap, adjust the flow to about 500 mL (1 pint) per minute (approximately 1/8-inch diameter stream).
6. Add preservative if required.
7. Fill the bottle to the shoulder about 1/4 inch from the top of the bottle.
8. Place the cap on the bottle and screw the cap on the top.
9. Complete the necessary forms with the appropriate information such as Public Water System (PWS)

identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, in distribution system). Also mention the type of analysis to be run. Complete a **COC** Record.

10. Pack the samples with ice to lower the temperature to 4°C. Ship to the laboratory by overnight courier or deliver to the laboratory.

Results may be reported as nitrate/nitrite, nitrate only, or nitrite only. When nitrite and nitrate need to be determined separately the holding time of 48 hours for nitrite must be met.

9.5.5 Other Primary and Secondary Contaminants. The primary drinking water standards relate to contaminants with health affects and the secondary drinking water standards are for contaminants that affect taste, odor, and appearance of the water. In some states and local municipalities, the secondary drinking water standards are monitored for compliance.

Primary contaminants include asbestos, barium, cadmium, chromium, copper, fluoride, lead, mercury, nitrate, nitrite, and selenium. Some of these contaminants have been discussed above. Secondary contaminants include aluminum, chloride, color, copper, corrosivity, fluoride, foaming agents, iron, manganese, odor, pH, silver, sulfate, total dissolved solids, and zinc. These samples are collected from the entry point to the distribution system or within the distribution system. 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 every three years for ground water and every year for surface water supplies.

Waivers are available for asbestos if it is determined that your system is not vulnerable to contamination (you do not have asbestos lined pipes or naturally occurring asbestos in the area, etc.). Contact the state or Federal agency that regulates your system for the proper forms to submit and to see if waivers for other parameters are available for your system.

Sampling Containers. The containers used for sampling are specified by the methods that will be used for analysis. Sample containers may be glass or plastic with volumes ranging from 50 mL to 1 liter. Most inorganic contaminants are sampled using a one-liter plastic container. Some of the parameters may be combined and sampled in the same container if the preservation and sample handling are equivalent. Combining the parameters with like preservations allows the number of containers sent to the laboratory to be reduced.

Sampling Procedures. The general sampling procedure should be used for collecting the sample as found in **Section 9.5.4**, Nitrate/Nitrites. The method of filling the container may be similar to microbiological sampling or specially designed to meet the data quality needs of the user of the data. The bottles are not usually filled to the top, but should be filled to allow air space in the top for sample mixing prior to removing the sample for analysis. The container used for drinking water samples should be free of the contaminants of interest at the level of detection. The FSP should address the container size, material, quality, and method of filling. Refer to **Appendix H** if the FSP is insufficient. Additional information about the containers, preservation, and filling is available from the laboratory.

The preservative is added to the container in the field, in the laboratory or, in the case of metals, may be added after the sample is sent to the laboratory. Chemicals used for preservation include nitric acid, sulfuric acid, and sodium hydroxide. The chemical used for preservations should be checked prior to use to ensure no contaminant is present at the level of detection. Preservation requiring a specified pH level should be checked with a pH meter or litmus paper, after addition of the preservative, and noted as to the amount of preservative, final pH, date, time, and person preserving the sample. The samples should be cooled to 4°C. Samples for metal analysis are not required to be cooled.

Holding times vary depending on the parameter being measured. Holding times are calculated from the date and time sampled to the date and time analyzed. Holding times range from 48 hours to six months. As with other kinds of samples, collectors should check with the regulator or approved methods to make sure they are using the correct preservation methods.

The methods approved for drinking water testing list the information necessary for preservation, sampling, and quality control. For secondary contaminants, the extent of method and field quality control is dependent on data needs. In most cases, quality control is limited to proper performance of the method and does not include field QC samples, such as duplicates and blanks. The data use, regulation or method will determine the extent and need for quality control samples.

9.5.6 Volatile Organic Compounds (VOCs). Many organic compounds have been found in drinking water (surface and ground water supplies). Contamination can be due to leaking underground storage tanks, landfills, wastewater disposal, injection wells, pesticide applications, etc. Samples for VOCs are collected from the entry point to the distribution system after treatment. Composite sampling is allowed but must follow very specific sampling procedures. The separate grab samples are taken by the sampling personnel, but the compositing must be performed by a certified laboratory at the time of analysis (Refer to state program prior to performing any compositing). The parameters included in VOC monitoring are both regulated and unregulated parameters. A complete list of VOCs can be found in 40 CFR 141.40.

Trihalomethanes (THMs). Trihalomethanes are also VOCs. Monitoring is required for populations of over 10,000. THMs are a result of chlorination and are produced during the treatment process. The formation of these compounds depends on the precursor concentration, chlorine dose, contact time, and pH. Unlike other VOCs, samples are collected within the distribution system after treatment. The four compounds of concern are chloroform, bromoform, chlorodibromomethane, and bromodichloromethane.

Sample Containers. The laboratory normally supplies the containers, preservatives, and blank water for VOC sampling. Recommended containers are glass, 60-to-120 mL screw cap vials with a Teflon$^{®}$-faced silicone septum. The preservatives, containers, and blank water should be traceable and controlled to ensure no detectable VOCs are present. Positive results in samples may result in increased monitoring and public notification.

Trip Blank. Trip blanks (or transport blanks) are to be prepared by the laboratory. These will be two lab filled vials in each shipping container used to ship water samples. The trip blank is used to show that contamination does not occur during transportation of the samples to the lab.

Field Blanks. Field blanks are samples of reagent that is transferred from one vessel to another at the sample site. The reagent used for the field blanks should be prepared by the laboratory by filling sample bottles with ASTM Type I or II water, sealing and shipping to the sampling site along with the empty bottles. This blank is used to show that the atmosphere at the sampling point has not caused contamination.

Sample Preservation. For collecting chlorinated samples, a dechlorinating agent such as 25 mg ascorbic acid should be pre-added by the lab supplying the sample containers. If gases are not to be determined, sodium thiosulfate may be used as the dechlorinating agent. It should rapidly dissolve as the bottle is filled. For samples that require HCl, add one drop of 1:1 HCl (one part acid to one part of volatile organic free water) to each 20 mL of sample volume. Sample pH should be less than 2 after adjustment. Table 9-5 gives a summary of recommended preservation for different VOCs.

Table 9-5 Recommended Preservation for Volatile Organic Compounds*

Constituents	Chlorinated	Non-chlorinated
Halocarbons & Aromatics	HCl + reducing agent	HCl
THMs	Reducing agent (HCl optional)	None required
EDB/DBCP	None required	None required

*From Standard Methods for the Examination of Water and Wastewater, 18th edition.

Sampling Procedures. Collect all samples in duplicate and prepare two field blanks for each sampling site. Collect **VOC** samples as follows:

1. Determine the appropriate representative sampling location. For a surface water system, the plant tap after treatment, as the water enters the distribution system may be appropriate. For a well discharging directly into the distribution system, a tap on the well's discharge piping, after any treatment, may be appropriate. For THM, a point within the distribution system will be selected.
2. When sampling from a tap, open the tap and allow the system to flush until the water temperature has stabilized (usually 10 min.). Remove hoses and other sources of contamination from around the tap prior to sampling. Adjust the flow to about 500 mL (1 pint) per minute (approximately 1/8-inch diameter stream).

WARNING

Concentrated acids and other chemical preservatives represent a safety concern. Refer to safety plan or HASP, as appropriate to scope of project, and Chapter 3, Sections 3.33 for precautions and personnel protections equipment. (PPE).

3. 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.
4. When sampling from an open body of water, fill a clean, empty (no preservative), one quart, wide-mouth bottle with sample and carefully fill two sample vials from the wide-mouth bottle. (Discard water in wide-mouth bottle after filling vials.)
5. 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 complete filling the bottle.
6. When filling the bottle, form a meniscus (the curved upper surface of a liquid formed by surface tension) or use the vial cap to top off the bottle and form a meniscus.
7. 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.
8. 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.
9. **CAUTION**

Air bubbles larger than approximately 1 mm can invalidate the VOC sampleGlass vials must be filled completely and handled with care to prevent spillage.

10. Fill out the label in waterproof ink. Be sure to clearly identify the exact sample collection location, date, and time of collection. If the sample collection point has a specific coded identification, include it on the label and sample submission form.
11. Repeat steps 6 through 12 for duplicate and field blank samples.
12. Complete the necessary forms with the appropriate information such as Public Water Supply (PWS) identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap entry point, and at distribution). Also mention the type of analysis to be run. Complete a **COC** Record.
13. Pack the samples and blanks with ice to lower the temperature to 4°C. Ship to the laboratory by overnight courier or deliver to the laboratory.

9.5.7 <u>Pesticides and Synthetic Organic Chemicals (SOCs)</u>. 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. The parameters to be measured are based on the size of the supply, use of the materials in the area and state discretion. When initial monitoring indicates results below the MCL, repeat monitoring is reduced to two samples every three years for supplies serving a population of more than 3,300 and to one sample every three years for supplies serving a population of less than 3,300. The state establishes the parameters, frequency, and timeline for monitoring for each water supply.

The methods used for monitoring the SOCs must be approved for drinking water monitoring due to the low level of detection required. Screening methods for PCBs are listed in the approved methods. The methods include the quality control samples required for the laboratory and field operations. The FSP must reflect the selected method requirements for sampling, preservation, holding times, and quality control samples.

Depending on the specific pesticide and chemical to be analyzed, the sample container and preservative chemical will vary (See **Appendix H**). 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 4°C during transportation and storage. Chlorinated and nonchlorinated water supplies are preserved using different preservatives. Preservatives include sodium thiosulfate to remove chlorine, hydrochloric acid to stabilize the pH and reduce biological activity, and other chemicals such as mercuric chloride. The use of mercuric chloride requires samples and waste material to be treated as a hazardous waste for mercury.

Sample Containers. 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, one-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. The general sampling procedure should be used for collecting the sample as found in **Section 9.5.4**, Nitrate/Nitrites.

9.5.8 Radionuclides. Samples for radionuclides are collected once every four years unless more frequent sampling is required by the state. Initial monitoring is performed by collecting a year long composite, one sample per quarter. Repeat monitoring may be reduced to a single grab sample, if approved by the state.

If gross alpha particle activity is monitored and is found to be above the MCL, additional monitoring for radium-226 and radium-228 may be required. If the water source is near nuclear facilities, additional parameters (e.g., Tritium, Strontium-89, Strontium-90, Iodine-131, and Cesium-134) may need to be monitored. These additional parameters will be specified in the state or Federal permit.

Preservations for the 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 may be added upon arrival at the laboratory. The preservative must be added within 5 days of collection and analysis must not be started until 16 hours after acidification. Preservatives must be checked for the absence of the radionuclides being monitored. Holding times are not directly addressed in the rule, but are commonly accepted to be six months, the same as samples for metals testing.

Sample Containers. Radionuclide samples should be collected in a plastic or glass container. The exception is Tritium, which must be collected in glass.

The size of the container is dependent upon the volume of sample needed to meet the required MCLs and the total dissolved solids present. Sample volumes will range from 0.5 liters to 18 liters. Sample volumes to be collected must be confirmed with the laboratory performing the analysis.

Sampling Procedures. The general sampling procedure should be used for collecting the sample as found in **Section 9.5.4**, Nitrate/Nitrites.

The methods currently promulgated in 40 CFR 136 are from Standard Methods for the Examination of Water and Wastewater, 18th edition, although the 19th edition is available. The Field Sampling Plan should include method references, preservation, containers, holding times, and sample size.

9.5.9 Sodium and Corrosivity. One sample per plant should be collected at the entry point to the distribution system for sodium and corrosivity. The number of samples is based on the number of treatment plants used by the system.

Sodium. The **Secondary Maximum Contaminant Level (SMCL)** for sodium is 20 mg/L. Samples must be collected annually for surface waters and every three years for groundwater sources. In areas where the sodium content is variable, more frequent analyses may be required.

Corrosivity. Determination of the corrosivity characteristics of the water includes measurement of pH, calcium, hardness, alkalinity, temperature, total dissolved solids, and calculation of the Langelier Index. Only one round of sampling is required unless the state specifies more. For surface waters, two samples per plant shall be collected, one during mid-winter and one during mid-summer. For groundwater sources, one sample is required for each plant. More samples and parameters may be required by the state.

9.5.10 Surface Water Treatment Rule. This rule, effective December 30, 1990, requires water treatment in lieu of water testing because the contaminants are difficult to detect and pose acute health risks. Disinfection and filtration for surface water and groundwater under the influence of surface water are required if microbiological, turbidity, and other standards in the rule are not met. Monitoring of disinfection and filtration treatment techniques is performed by measuring coliform density, heterotrophic bacteria, chlorine residual, and turbidity.

In some large supplies (populations greater than 100,000) and some states, sampling and testing for microorganisms is being conducted. The sampling methods, test procedures, and results interpretation should be performed only by individuals specifically trained and experienced with the organism for which tests are being performed (e.g., Giardia Lamblia and Legionella).

Bacteria monitoring. Bacteria sampling for coliform is performed in the same manner as described in **Section 9.5.2.** The reporting for bacteria testing for complying with this rule requires samples to be collected at the source prior to treatment. The results reported must be numeric and not presence/absence as in the Total Coliform Rule.

The methods acceptable for bacteria testing differ from distribution system monitoring due to the reporting requirements of the Surface Water Treatment Rule. 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 in 40 CFR part 141.

pH, temperature, and chlorine residual monitoring. The sample should be representative of the disinfection system. The sample should be collected at the point of entry after disinfection. pH, chlorine and temperature field measurements procedures are presented in **Chapter 14**. The chlorine residual should be 0.2 mg/L minimum at all times.

Turbidity monitoring. The turbidity sample should be representative of the system's filtered water. The regulator should specify where the turbidity samples must be taken for compliance. The turbidity after conventional or direct filtration should be less than 5 Nephelometric Turbidity Unit (NTU) and less than 0.5 NTU in 95% of all samples collected. Slow sand and diatomaceous earth filtration systems must achieve a filtered water turbidity level of less than or equal to 1 NTU in the 95% of the measurements of each month (limits may vary by state). At no time can the filtered water turbidity exceed 5 NTU. Samples should be collected once per day.

Collect the sample in a clean glass or plastic one-liter bottle from the designated sample collection point. Samples should be analyzed on the same day collected or stored in the dark for a maximum of 24 hours. If samples are transported or stored prior to analysis, pack samples with ice to lower the temperature to 4°C.

Complete the necessary forms with the appropriate information such as Public Water Supply (PWS) identification number, exact sample collection location, date and time, and type of sample (e.g., raw, tap, entry point, in distribution system). Also mention the type of analysis to be run. Complete a COC Record.

The method used for measuring turbidity must use the standards and quality control required in the approved methods (Refer to **Appendix H**). Only turbidity meters meeting the requirements of the approved methods should be used. 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.

9.6 **<u>QUALITY CONTROL / QUALITY ASSURANCE (QA/QC)</u>.** The following protocol should be used to ensure integrity and accuracy of the data collected during drinking water sampling. The laboratory analysis must be performed by a state certified laboratory for the method required.

All samples must be accompanied by a complete **COC Record**. Field Blanks, Trip Blanks, and Field Duplicates may be collected as part of 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. See **Appendix I** for required and recommended QC sample(s).

The main elements of the Quality Assurance Plan (QAP) for drinking water sampling are:

- QA objective for measurements
- Sampling procedures
- Sample custody
- Calibration procedures

- Analytical procedures
- Preservation techniques
- Lab performance evaluation samples
- Performance audit
- Data assessment or validation, if necessary
- Quality assurance report
- Corrective actions

9.7 SAMPLING EQUIPMENT LIST. Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to drinking water sampling:

☐ Thermometer
☐ Litmus Paper or pH meter
☐ Field Blanks
☐ Waterproof pens (blue or black)
☐ Sample container labels
☐ Clear wide plastic tape to cover sample container labels and shipping labels
☐ Shipping container and materials such as:
  ☐ Clear plastic resealable food bags for sample containers, and COC
  ☐ Ice

Additional equipment needed for sampling by constituent:

Chlorine
☐ Chlorine Test Kit or Diethylphenylenediamine (DPD) Test Kit

Total Coliform/ Bacteria
☐ 120 mL Plastic or Glass Containers (sterilized)
☐ Sodium Thiosulfate - a preservative

Copper and Lead
First Draw
☐ Liter Bottle, Plastic or Glass
☐ Nitric Acid - preservative
☐ Warning Signs - to notify potential users to not turn on system during testing period

Service Line Samples
☐ Pitcher or Bucket
☐ Sampling Tap
☐ Liter Bottle Plastic or Glass
☐ Nitric Acid - preservative

Nitrates/Nitrites
☐ Two 1 Liter Plastic or Glass Bottles
☐ Sulfuric Acid (For Nitrate non-Chlorinated water Only)

Inorganics
☐ Liter Plastic Bottle
☐ Preservative (Check FSP for specific preservative or see **Appendix H**)

VOCs
☐ mL Screw Cap Vials with Teflon faced Silicon Septa
☐ Dechlorinating Agent (Ascorbic Acid)
☐ HCl - if sampling for halocarbons or aromatics
☐ One Quart Wide Mouth Bottle

Pesticides and Synthetic Organic Chemicals
☐ Sodium Thiosulfate - if chlorine is present
☐ HCl - a preservative
☐ Mercuric Chloride - a preservative
NOTE: Samples will be a hazardous material
Liter Amber Glass Bottles with Teflon-lined Lids

Radionuclides
☐ HCl - if sampling for Cesium-134
☐ Nitric Acid - for all others except Tritium and Iodine-131
☐ Plastic or Glass Bottles - volume dependent upon sample volume

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 10

AIR SAMPLING

10.1 PURPOSE. This chapter provides supporting background information and general procedures for air sampling related to compliance issues.

10.2 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.

10.3 BACKGROUND. Air sampling is conducted to monitor levels of air contaminants. Representative air sampling presents unique and difficult challenges. Air is the most difficult environmental matrix to sample representatively because contaminant concentrations are affected by many variables which are subject to rapid and often drastic changes. Also, the contaminants of interest may be present at very low concentrations.

10.3.1 Air Matrices. Air sampling matrices include:

- Indoor air
- Ambient (outdoor) air
- Point sources (stacks, exhausts, and other emission sources)
- Fugitive emissions (sources of air pollutants other than stacks or vents)
- Soil atmospheres, particularly over and around landfill waste sites

Figure 10-1 shows expected air pollution concentration ranges for various air matrices.

10.3.2 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

Analytically, the basic objective of air monitoring is to measure representatively *insitu* or transfer all of the contaminant from a measured quantity of air into an analytical instrument, which 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 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
- 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 compared 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 limits, data turnaround needs, data quality objectives, and regulatory requirements written into facility operating permits.

10.4 GENERAL CLASSIFICATION OF AIR POLLUTANTS. Generally, air pollutants are broadly classified into two types - **gases** 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.

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 compound

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 particle size range. Aerosols can be formed by:

- water vapor (i.e., fog, steam)
- smoke and industrial fumes
- reaction of species such as sulfur dioxide with water
- growth of smaller entities such as salts into agglomerates (i.e., ammonium sulfate and nitrate)
- organic liquid particulates from the distillation or vaporization of volatile materials
- Atomization of liquids through a small orifice

There are numerous types of solid particulates:

- **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** - non-combustible mineral solids formed from complete oxidation of coal or fuel oil, generally <200 micron diameter
- **dusts** - particulates from natural or industrial disintegration processes, 1-1000 micron diameter
- **fumes** - particulates from condensation of distillation vapors, calcination, or chemical reactions

Liquid particulates include:

- **mists** - particulates arising from vapor condensation, chemical reactions, or liquid atomization (i.e., steam or fog)
- Chromic acid mist from electroplating

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 particulate

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 adsorbed to solid particulates and several soluble aerosols co-dissolved. **Figure 10-2** illustrates relative particle size ranges for aerosols, dusts, and fumes.

Aerosols are a primary concern in air pollution because they result in reduced visibility due to their light scattering and light absorption properties. Smoke and ammonium sulfate aerosols are particularly deleterious to visibility. The meteorologic definition of visibility refers to the measurement of the greatest distance at which a dark object of reasonable size may be seen against the horizon sky. This measurement depends on the degree of light transmission through the atmosphere and the contrast of the object to the background. Generally, gases do not affect the transmissive properties, but occasionally, nitrogen dioxide (NO_2) may be present in sufficient concentration to create a yellowish-brown discoloration. The aerosol particles most impacting visibility are in the 0.3 - 0.6 micron range. Measurements of visibility are usually made with transmissometers and often use photographic images which are computer analyzed for contrast data.

10.5 FACTORS AFFECTING AMBIENT AIR SAMPLING AND MONITORING.

10.5.1 Meteorological.

10.5.1.1 Meteorological Parameters. Primary meteorological parameters to be considered in ambiet air sampling are:

- Wind Direction
- Wind Speed
- Temperature
- Atmospheric stability
- Atmospheric pressure
- Precipitation

Wind direction is most critical and must be monitored an 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-volatile by increasing the amounts absorbed by particulate such as soil and dust. Increased wind speed facilitates transfer of a greater number of large particulate from the source.

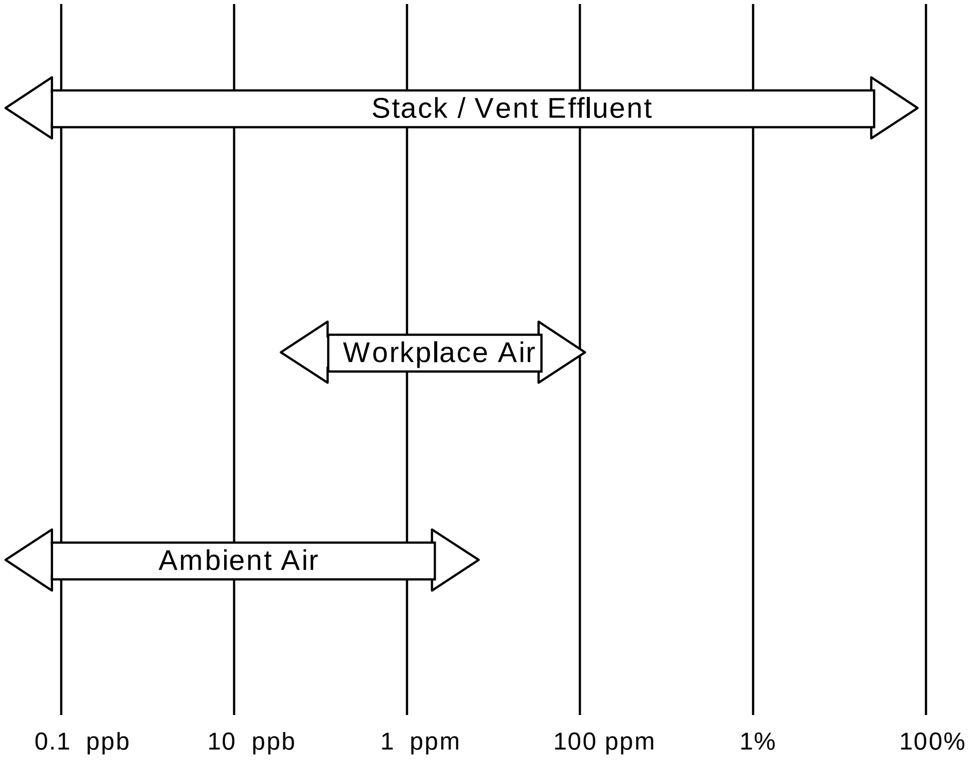


Figure 10-1 Expected Air Pollutant Concentration Ranges for Various Air Matrices

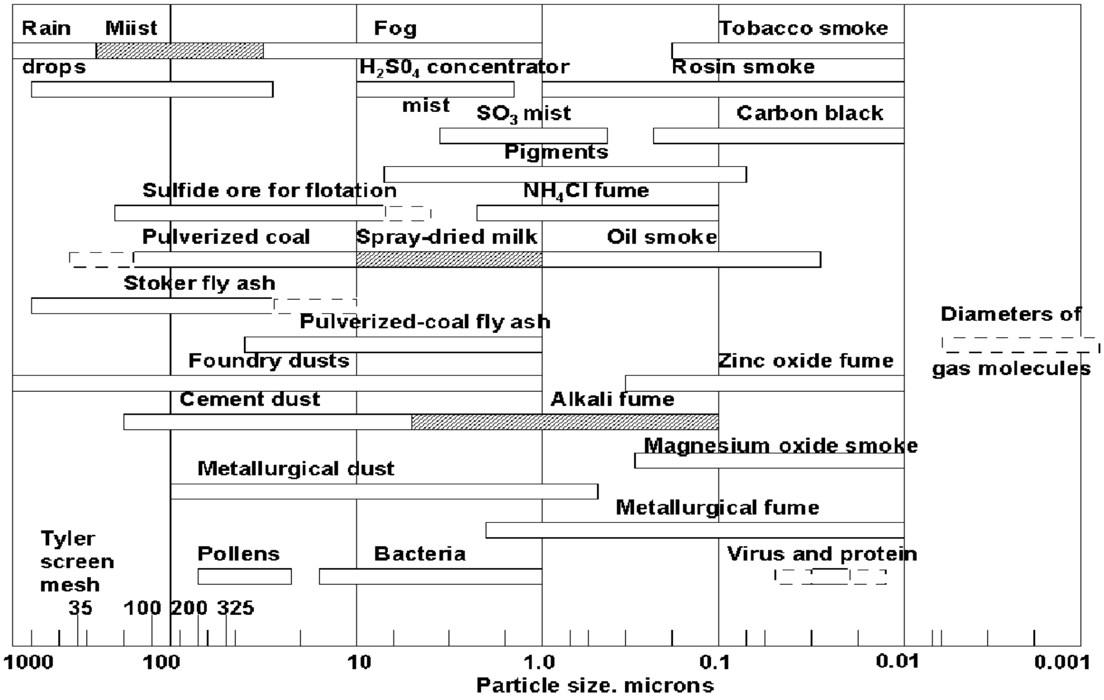


Figure 10-2 Particle Size Ranges for Aerosols, Dusts and Fumes

Source: Faith, W. L. and Atkinson, A. A., Jr.

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% humidity also significantly decreases particle transport by wind and volatilization rates.

10.5.1.2 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.

10.5.1.2.1 Wind Measurements. Variable heating and cooling of the earth's surface is the primary cause of wind, water and climate. Uneven heating results is hotter air near the equator and colder air near the poles. A circulation pattern develops as hot air flows to the poles and colder moves towards the equator. The rotation of the earth on its axis results in the Coriolis force which causes air flows in opposite directions in the northern and southern hemispheres. The result is three bands of air flows in each hemisphere. Near the equator and the poles, winds are generally from the east. At the mid-latitudes they are generally from the west. These are, however, only average conditions since winds rarely come from one direction for long periods, especially in the mid-latitudes. Seasonal trends localized winds such as the Kona winds and "nor'easter" winds modify the generalities.

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, or a height above ground equal to the height of the stack or source being measured. It is very important that the structure supporting the monitoring equipment not in any way interfere with the wind sensors, 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. For some models, wind direction standard deviation may be used.

Either prop and vane or rotating cup type 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.

10.5.1.2.2 Temperature and Humidity. For the purposes of most modeling, temperature and humidity measurements can be made which are representative of hourly periods. 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 build up 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.

10.5.1.2.3 Total Incoming Solar Radiation. Total incoming solar radiation includes both direct and diffuse radiation. One instrument for measuring solar radiation is a pyranometer which consists of a surface with a series of thermojunctions painted optical black and reference junctions painted white. When solar radiation strikes the surface, a temperature difference is recorded. The junctions are protected from the atmosphere by a glass dome.

10.5.2 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.

10.5.3 Atmospheric Turbulence. Atmospheric turbulence refers to the various sized swirls of motion (eddies or eddy currents) present in the atmosphere. It is the combined result of mechanical mixing due to irregular air flow over roughness elements on the surface of the earth, and buoyant effects resulting from heating and cooling of the earth's surface and surrounding air masses.

10.5.3.1 Mechanical Turbulence. Mechanical turbulence occurs as air flows over rough or irregular surface elements. Roughness of surface features largely determines the size of eddies and their effective atmospheric height. Forests, mountains, and high rise cities create large eddy turbulence, while turbulence over plains and other flat areas is generally small. High wind speeds over rough surface elements also create high turbulence. Mechanical turbulence can also be generated from shear motion as a faster moving air mass moves past a slower one. Near the ground where air flow is slowed by surface friction, shear forces are common.

10.5.3.2 Buoyant Turbulence. Buoyant turbulence is the result of warm air rising and being replaced by cooler downward moving air. The warm air results from UV radiation heating the ground which then heats the surrounding air through convection from the ground and radiant heat in the form of infrared radiation. Buildings, parking lots, industrial processes, fuel combustion, and heating and air conditioning systems contribute greatly to positive buoyant turbulence. At night, replacement of warm air near the ground by cooler air results in negative buoyant turbulence. A simplified model of buoyant turbulence is a slow-motion picture of gently boiling water.

10.5.4 Thermal Gradients. Due to heating and cooling of the earth's surface and surrounding air layers, and the decrease of atmospheric pressure with vertical height, vertical temperature gradients result. Conventionally, if temperature decreases with height, the vertical temperature gradient is negative. If temperature increases with height, the gradient is positive.

Positive gradients are called "inversions" and are considered very vertically thermally stable. Negative gradients form vertical columns of rising air called "thermals". The rising air is replaced by sinking surrounding air creating a swirl effect. This is considered to be an unstable condition.

The magnitude of temperature change over the vertical height compared to a standard heating/cooling rate of about 0.01^{o}C per meter in dry air is used to evaluate atmospheric vertical thermal stability. The standard heating\cooling rate for dry air is called the dry adiabatic lapse rate. Vertical temperature differences more positive than the dry adiabatic lapse rate indicate vertical thermally stable atmospheres. Those less positive are considered unstable.

The effects of thermal gradients and altitudes on plume shapes from continuous release sources are shown in **Figure 10-3**.

10.5.5 Atmospheric Layers.

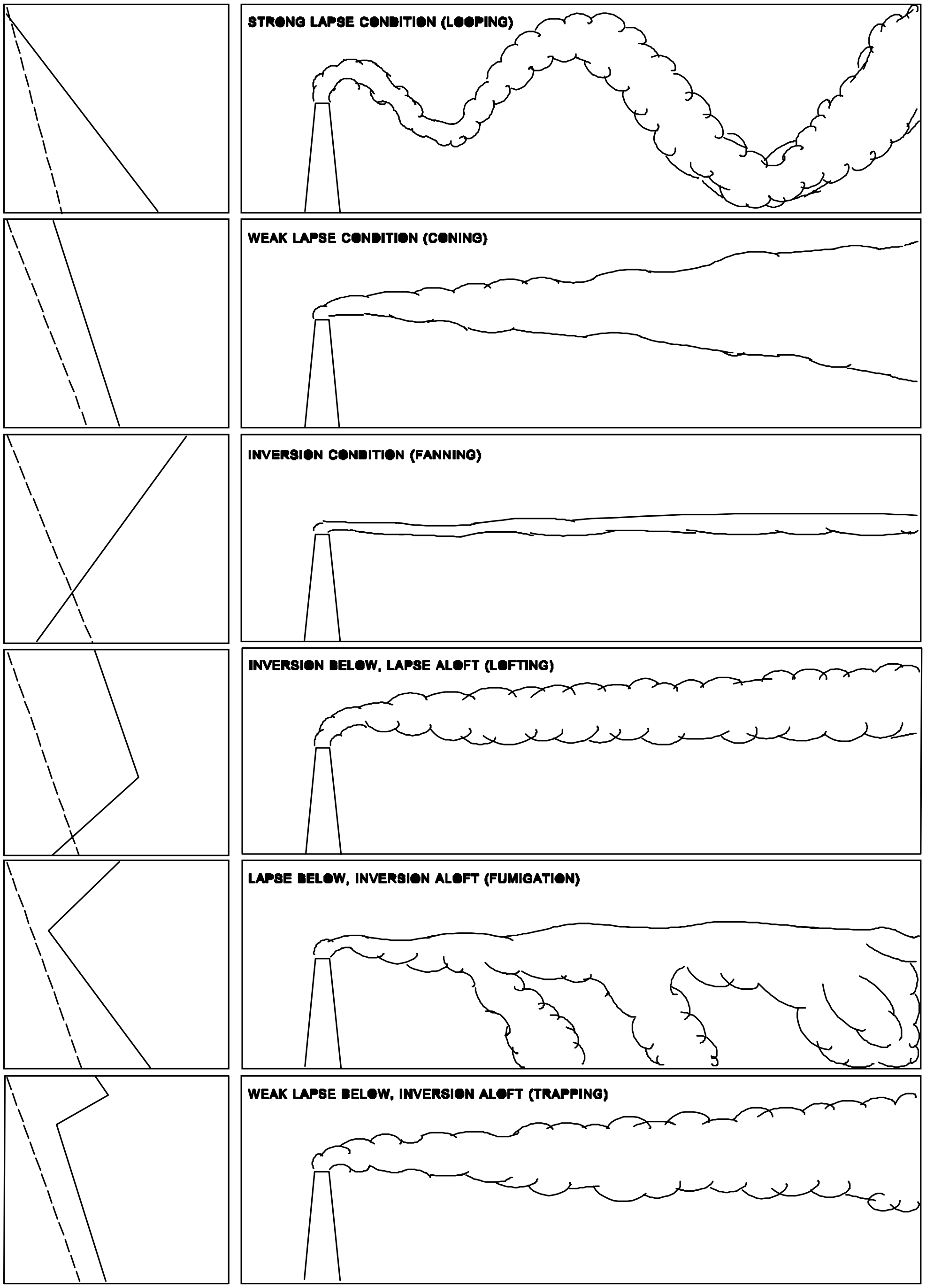
10.5.5.1 Boundary Layer. The layer of atmosphere influenced by ground conditions is known as the boundary layer.

10.5.5.2 Gradient Wind. The free flowing wind above the boundary layer is the gradient wind.

10.5.5.3 Mixing Layer. The top of a neutral or unstable surface air layer is called the mixing layer. For stable surface air layers such as inversions, the mixing layer is undefined.

10.5.6 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. Minute incidental contaminants on the large surfaces of containers and sorbents used to collect or concentrate samples can also pose significant problems. Because inadvertent contamination, it is imperative that all sampling equipment be scrupulously clean and many types of blanks run. Sampling and sample handling procedures must also be meticulous to detail.

10.5.7 Time Resolution of Sensors. Time resolution is the time required for a real-time sensor, recording *insitu* measurements, to reach 90% of the final



Altitude versus Temperature
Legend:
---- Dry Adiabatic Lapse Rate
__ Actual Lapse Rate

Figure 10-3 Effects of Thermal Gradients and Altitudes on Plumes from Continuous Release Sources to an incremental change in contaminant concentration. This is an important criteria to consider when selecting monitoring equipment for a specific application.

10.5.8 Placement of Samplers and Monitors. Siting 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.

10.6 AIR SAMPLING GENERAL TERMS.

10.6.1 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 either a filter device, a trapping liquid, or a solid sorbent media.

10.6.2 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 which collect samples for analysis in the 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.

10.6.3 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.

10.7 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.

10.7.1 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

Depending on the type of source, the concentration of emitted gas is measured from the emitting surface, or from a source outlet line. From a variety of parameters, the relative concentration, or emission flux is determined. The emission flux is the rate of emission/area/time. Combination of measurements from several monitoring sites can be used to determine emission rates for the overall site. It is advisable that real-time monitors also be used and the data correlated with the flux chamber data. This method has several advantages:

Advantages:

- cost effective
- simple equipment requirements
- all measurements direct
- no models or estimates required
- generally better quality data

- no upwind contamination and few meteorological interferences

Disadvantages:
- not always possible
- primarily used for volatiles materials
- must exercise caution in data extrapolation

10.7.2 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 weighing 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:
- 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:
- 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

10.7.3 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-type 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** 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:
- can be done at greater distance from the source
- exact data on source emission not required

Disadvantages:
- 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

10.7.4 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:
- fast
- inexpensive
- requires little or no field measurement
- useful screening tool

Disadvantages:
- provides estimate only
- fit of site source and model critical
- can be very inaccurate

10.8 FIELD SAMPLING INSTRUMENTS/ EQUIPMENT.

10.8.1 Field Measurement Instrumentation. Instruments have been developed for field operations to allow real-time measurement of air quality. Most are meters which measure analyte concentrations by passing a stream of air through the detector for a finite time interval. Others monitor continuously and sound an alarm when a pre-set concentration level is reached, 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:

- All field instruments have 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, many 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:

- Immediate readout
- Portability

Disadvantages:

- Response usually is not specific to a single chemical
- Detection limits are generally in the range of acute health effects (i.e., instruments may not have the sensitivity needed to measure low concentrations of pollutants)
- Relatively expensive compared to 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.

10.8.1.1 Volatile Organic Chemicals (VOCs). VOCs include 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, response should depend on the type and concentration of VOCs present. **Table 10-1** is a list of commercially available portable VOC detection instruments.

10.8.1.1.1 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 10-4**). 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 has units of parts-per-million (ppm), which should be interpreted as the equivalent response to the calibration gas, not the ppm of unknown VOCs in the air. **Table 10-2** lists the relative response of a PID instrument to different organic chemicals in air.

Advantages:

- Stable, calibration remains fairly constant
- Easier to ship, no flammable gas is required

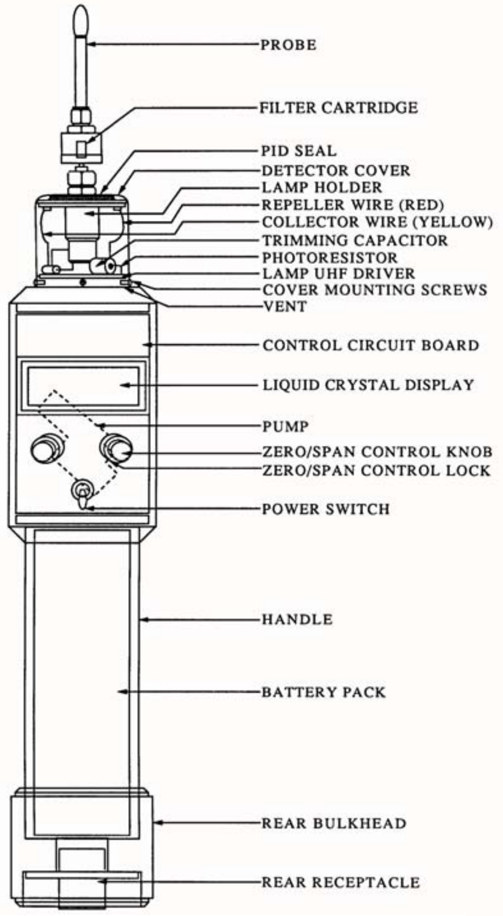


Figure 10-4 Photo Ionization Detector

- Sensitive to commonly encountered VOCs, such as gasoline
- Not affected by presence of methane in air

Disadvantages:

- Sensitive to high humidity
- 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

10.8.1.1.2 **Flame Ionization Detector (FID).** A FID works by using the contaminated air to burn hydrogen in a flame within the instrument. When most organic vapors burn the residue 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. The relative instrument response to different VOCs is summarized in **Table 10-3**.

Advantages:

- Response is less a function of the particular chemical than with the PID
- Less sensitive to moisture than the PID

Disadvantages:

- 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 parts per million VOCs in air

10.8.1.1.3 **Infrared Radiation Absorbance Detector.** Although VOC vapors are transparent to visible light, they will absorb infrared radiation. This detector passes ambient air through a beam of infrared light. The extent to which the different wavelengths of light 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:

- Specific for particular chemicals, since different VOCs have different infrared radiation absorbance characteristics

Disadvantages:

- Detection limit may be higher than the PID or FID (for instance, for xylene it is about 10 ppm)

10.8.1.1.4 **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:

- Inexpensive
- Direct readout

Disadvantages:

- 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 being purchased, depending on tube type

Table 10-1 Commercially Available Portable VOC Detection Instruments

Model number, Manufacturer	Detection Principle	Range: ppm	Sensitivity	Response time	Accessories	Calibration techniques	Applicable Methods
550, 551,555, 580 (AID, Inc.)	PID, FID	0-200 0-2,000 0-10,000	0.1 ppm at 0-200 ppm	<5 seconds		Bag sampling	
OVA 108, 128 (Century Systems Inc., Foxboro)	FID	0-10 0-100 0-1,000 0-10,000 0-100,000	0.2 ppm (Model 128) 0.5 ppm (Model 108)	2 seconds	Thermal desorbers available Optional GC available	Direct Injection Bag Sample	
TVA-1000 (Foxboro)	PID/FID Combination	ppm		2 seconds	Barcoding software	Gas cylinder	Method 21
Micro FID (PhotoVac)	FID	ppm		2 seconds	Bar coding software	Bag sample	Method 21
PI-101 (HNu Systems)	PID	1-20 1-200 1-2,000	0.1 ppm low molecular weight aromatics	<5 seconds	3 lamps available: 9.5 eV (aromatics) 10.2 eV (hydrocarbons) 11.7 eV (halocarbons)	External gas cylinder Bag Sample	
TIP (Photovac)	PID	0-2,000	0.05 ppm benzene	3 seconds		Bag sample	
Miran (Foxboro)	Infrared Radiation	ppm to %	1 ppm	1 to 40 seconds		Bag sample	
Detector tubes (Dr@ger; Sensidyne; Matheson)	Colorimetric tubes - length of color change	ppm	Depends on chemical	20 - 60 seconds	Different tubes available for many different VOCs and inorganic volatile chemicals		
ChromAir (Gilian)	Colorimetric badge - color change of sensor dots at various concentration ranges	ppm	Depends on chemical	8 hour	Different badges available for a limited number of VOCs and inorganic volatile chemicals		

Table 10-2 Relative Response of PID Detector

Chemical	Relative Response		Chemical	Relative Response	
	10.2 eV lamp	10.6 eV lamp		10.2 eV lamp	10.6 eV lamp
Acetone	0.63	0.452	Isopropyl alcohol	0.10	0.046
Benzene	1.00	1.000	Methane	0.00	0.000
Ethane	0.00	0.000	Methyl ethyl ketone (2-Butanone)	0.57	0.099
Ethylene	0.00	0.043	Propylene	0.40	0.454
Ethylene oxide	0.10	0.130	Toluene	1.00	0.814
n-Hexane	0.22	0.085	1,1,2-Trichloroethylene	0.89	0.734
n-Heptane	0.17	0.147	m-Xylene	1.12	0.900
Isobutylene	0.70	0.589			

Source: 10.2 eV: *HNu Operating Manual*. 10.6 eV: *Photovac TIP® Operating Manual*.

Table 10-3 Relative Response of FID Detector

Chemical	Relative Response	Chemical	Relative Response
Acetone	0.60	Isopropyl alcohol	0.65
Benzene	1.50	Methane	1.00
n-Butane	0.50	Methyl alcohol	0.15
Carbon tetrachloride	0.10	Methyl ethyl ketone (2-Butanone)	0.80
Chloroform	0.65	Methyl isobutyl ketone	1.00
Ethane	0.90	n-Pentane	1.00
Ethyl alcohol	0.25	Propane	0.64
Ethylene	0.85	Toluene	1.20
Ethylene Oxide	0.70		

Source: *Century Systems OVA Operating Manual*

10.8.1.1.5 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:

- Rugged
- Sensitivity to VOCs is in the percent range, which is relevant to explosive mixtures
- 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
- Not sensitive to the presence of VOCs at low levels that may still present toxicity hazards

10.8.1.2 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 testing air known to contain 10 ppm of H_2S.

10.8.1.3 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 are available which have sensors for explosive gas mixtures, oxygen, hydrogen sulfide, and CO. 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.

10.8.1.4 Particulates. The Occupational Safety and Health Administration (OSHA) permissible exposure limit for particulates 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:

- Real time data
- Portable

Disadvantages:

- Sensitive to fog and other aerosols
- Not size specific for dust particles
- Not easily calibrated in the field

10.8.1.5 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 heat cycle is manually activated to clear the accumulated mercury from the foil.

Advantages:

- 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:

- Not easily calibrated
- Quality control, via blanks and spike samples, is difficult in the field
- Specific only to atomic mercury fumes, vice organic mercury compounds which are even more toxic

10.8.1.5.1 Ozone. Ozone can be measured as a grab sample or by real-time continuous monitoring using established ultraviolet (UV) methods.

10.8.1.5.2 Nitrogen Oxides. In air free of aerosols and condensed phase atmospheric water, a variety of nitrogen oxide and oxyacid species can be analyzed using methods based on the chemiluminescent reaction of these compounds with ozone. Determination of NO_2 is not highly reliable because of the products of the catalytic conversion reaction used in the chemiluminescence method. Gaseous nitric acid can be determined by this method in real-time, or in 15-30 minutes by collection on filter packs on the surface of diffusion denuders.

10.8.1.5.3 Sulfur Dioxide (SO_2). Sulfur dioxide can be measured using flame photometric detection (FPD) after removal of aerosols by filtration onto materials that are SO_2 inert such as Teflon® or acid treated quartz.

10.8.1.5.4 Ammonia (NH_3). There are several methods available for measuring ammonia, but most have considerable limitations.

10.8.1.5.5 Computer Software and Bar Code Readers. Many equipment manufacturers offer computer software, bar code tags, and bar code readers to facilitate documentation and record keeping. Using these accessories, data can be logged by the instrument and downloaded for computer tracking systems. Hard copies of records can then be generated. Bar code systems are extremely useful when many emission sources must be tracked.

Advantages:

- More effective record keeping
- Less sampler time required

Disadvantages:

- Higher initial cost

10.9 SAMPLES COLLECTED FOR LABORATORY ANALYSIS.

Some samples can be collected and analyzed without field concentration. These survey samples are useful particularly for screening purposes. In other cases, the desired analytes are reactive compounds, present in very low concentrations. They may be distributed between two or more phases (gases, liquids, solids). Because most sampling devices disturb the phase equilibria, it is not always possible to determine analyte distribution between the phases. Since the analyte concentrations may be very low, it is generally necessary to concentrate the compounds of interest by filters (solid phase samples) or sorbent materials (gaseous or liquid phase samples).

The low detection limits required for ambient air data can generally be achieved only with sensitive laboratory instruments using complex analytical procedures. Performing analyses under controlled laboratory conditions also better supports the Quality Assurance program required to ensure the accuracy of data. 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.

10.9.1 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.

CAUTION

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.

CAUTION

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, and **Figure 10-5** illustrates various types of whole air samplers.

10.9.2 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:

- Light weight
- Easily transportable
- Inexpensive

Disadvantages:

- Short holding times: Leakage and/or permeation of chemicals into and out of the bag is a common 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 levels 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.

10.9.3 Glass Globe Samplers. Evacuated glass bulbs have been used extensively to collect whole air samples.

Advantages:

- 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:

- 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

10.9.4 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. Pre-evacuated, the canisters are used in one of two ways. **EPA Standard Sampling Method TO-14** presents detailed procedures for both sub-atmospheric sampling and pressurized sampling using evacuated canisters.

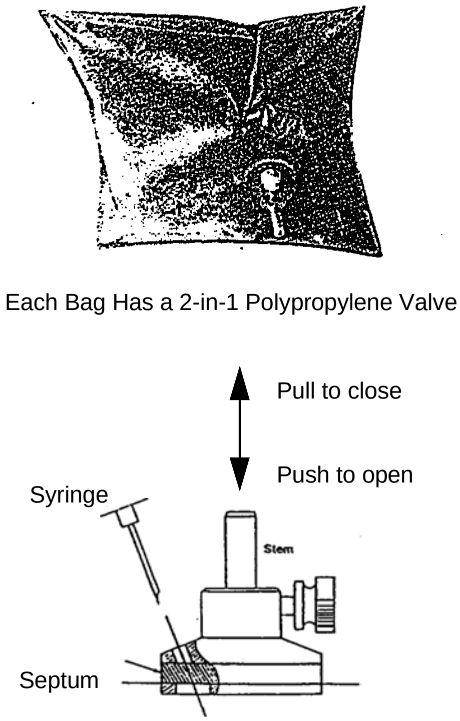
- **Sub-atmospheric sampling.** A canister is evacuated to 0.05 mm Hg. When opened to ambient air, the differential pressure causes air to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours) taken through a flow-restrictive inlet. A mass flow controller can maintain a constant flow rate from full vacuum to within about one psi or less below ambient pressure. With a critical orifice flow restrictor, a decrease in the flow rate occurs as atmospheric pressure approaches.
- **Pressurized sampling.** A metal bellows-type pump forces ambient air into an evacuated canister until pressure in the canister reaches 15 to 30 psig. For instance, a six liter evacuated canister can be filled at ten cm^3/minute for 24 hours to achieve a final pressure of about 21 psig. **This sampling technique is the TO-14 method most often used.**

Advantages:

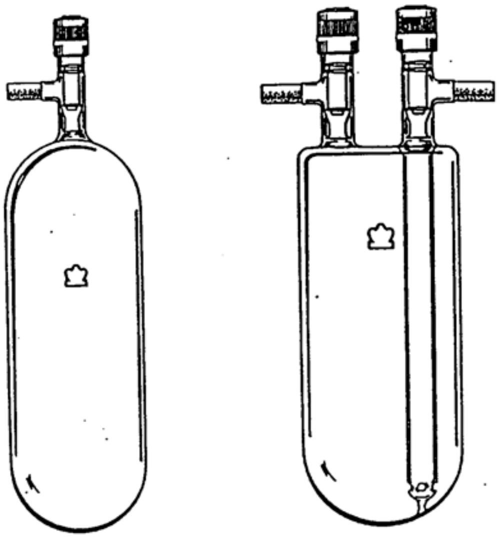
- Holding time: Canisters are not subject to sample permeation or photo-induced chemical effects (Sample integrity of trace level organic compounds stored in Summa® polished canisters has been demonstrated for up many chemicals for up to 30 days.)
- Reusable: Can be reused after a cleanup procedure
- Cleanliness: Generally, does not absorb contaminants, so it is relatively easily cleaned (It is, however, suggested that 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:

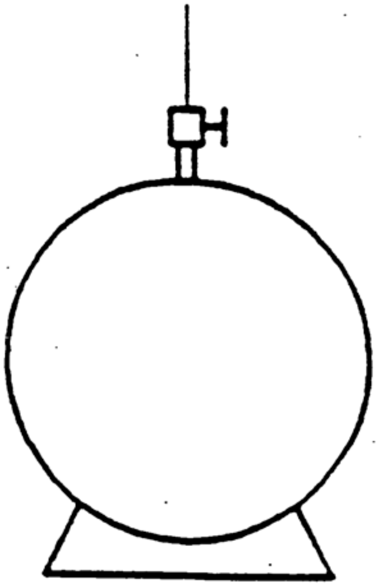
- HeavyExpensive



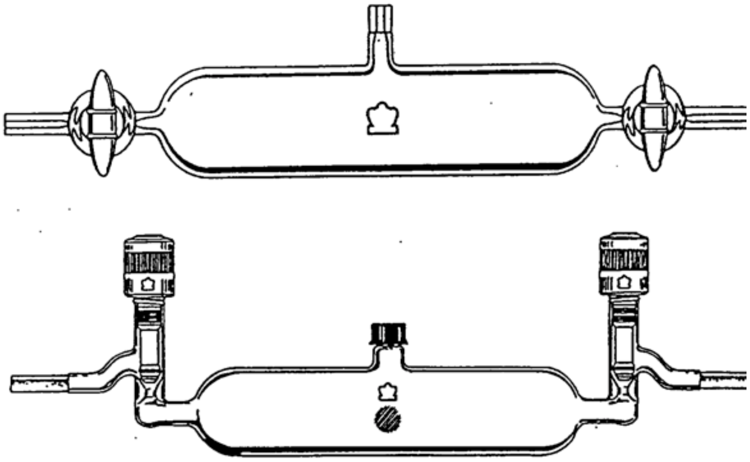
Teflon/Tedlar/Mylar/Saran Sampling Bags (with detail of sampling valve)



Gas Sampling Bottles



Stainless Steel SUMMA Canister



Gas Sampling Tubes

Figure 10-5 Whole Air Samplers

10.9.5 Sample Concentration Methods. The low detection limits required for ambient air data can be obtained by selectively removing the chemicals of interest from a measured volume of air and submitting 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 analysis 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, and **Figure 10-6** illustrates various air component concentration samplers.

10.9.5.1 Volatile and Semi-Volatile Organics. Methods of sample collection and analysis of organic compounds are driven primarily by the volatility and polarity of the compounds of interest. Volatile organic compounds (VOC's) have vapor pressures $>10^{-2}$ kPa. In general they are compounds with molecular weights less than C10 hydrocarbons. Semi-volatiles are in the vapor pressure range of 10^{-2} to 10^{-8} kPa. Non-volatiles have vapor pressures $<10^{-8}$ kPa. Various collection methods are used including adsorption on solid sorbent media, chemical reactions that form non-volatile products, and cryogenic freezing from the air. Generally, compounds with moderate to high vapor pressures are concentrated from a large volume of air on a solid sorbent material. Compounds with low vapor pressure are often associated with particulates, making some type of filtration followed by a vapor trap the preferred method of collection.

Very polar compounds are difficult to sample and analyze quantitatively. They adhere tenaciously to the walls of sampling containers and solid sorbent media.

Organic compounds in soil atmospheres can be sampled by boring small diameter holes in the soil to a depth of three or more feet. Plastic tubes with perforations at their base are placed into the holes. A peristaltic pump is then attached between the open end of tubes and a or Tedlar® sampling bag.

The following sub-paragraphs explain some methods used to collect and concentrate organic components from ambient air.

10.9.5.1.1 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 absorbing material is treated with solvents or heat to remove the chemicals of interest.

Advantages:

- 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:

- 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

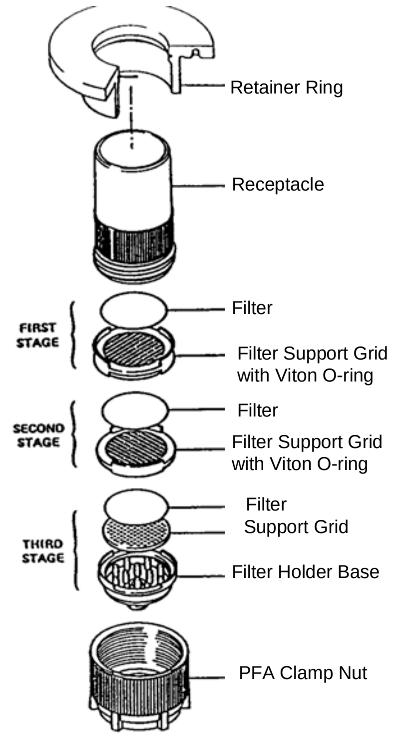
10.9.5.1.2 Active Air Samplers. These samplers use a pump to pull ambient air through the collection media. Sampling pumps are classified as high flow (500-3000 cubic centimeters/min) or low flow (50-200 cc/min). Flow rates should be maintained within +5% of set rate. Pump features may include:

- 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.

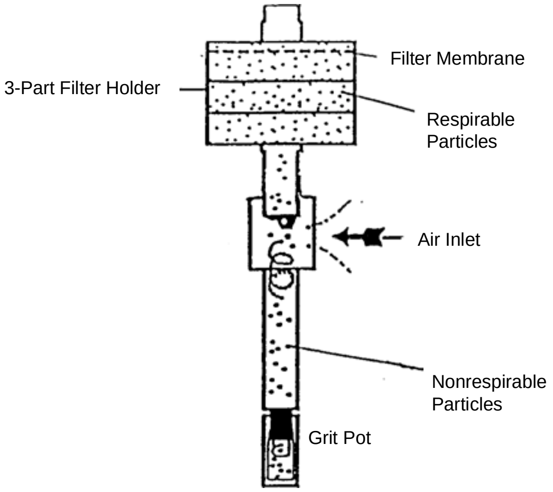
Various collection techniques are used to remove the chemicals of interest from the air. The EPA standard methods for sampling ambient air are listed in **Table 10-4**. For information on the use of the EPA sampling methods, see **Table 10-5**, *Methods TO-1 to TO-13* and **Appendix K**, *EPA Emission Test Methods Under Title 40.*

Advantages:

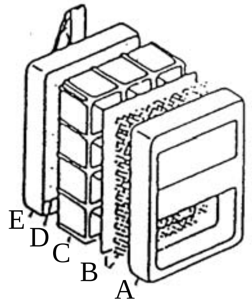
- Chemicals of interest are concentrated, resulting in improved detection limits
- Standard methods have been defined and validated
- Samples are collected over long periods to obtain data on average contaminant levels



3 - Stage Filter Pack
(Dry Deposition Sampling

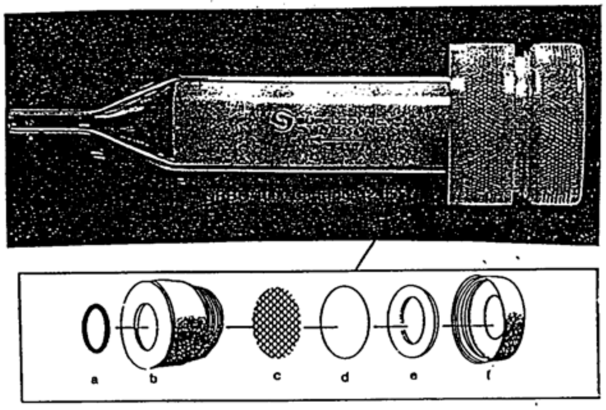


Air Cyclone Sampler



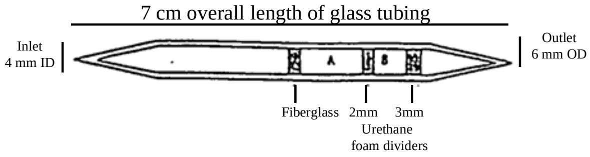
A - Sampler front
B - Draft shield
C - Grid section
D - Sorbant impregnated pad
E - Sampler back

Passive SamplingBadge



Supelco's Filter Cartridge Assembly, which contains a replaceable 32mm diameter quartz microfiber filter with stainless steel screen support, is attached to the front of the OR80-1000 PUF cartridge. The filter traps aerosol and particulate forms of semivolatilles, such as pesticides and PCBs. The low pressure drop of the filter cartridge, coupled with the OR80-1000 cartridge, allows high volume sampling at rates of 1-5L/min.
Several US EPA and ASTM methods require the use of a PUF (Polyurethane foam) absorbent cartridge with a optional quartz filter, combined the two sampling devices trap particulate aerosol, and vapor forms of semivolatile contaminates.

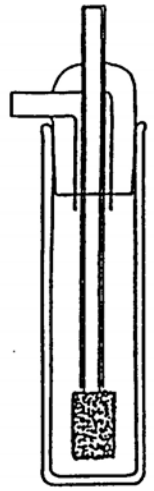
Polyurethane Foam (PUF)
Cartidge and Filter Cartridge
Assembly



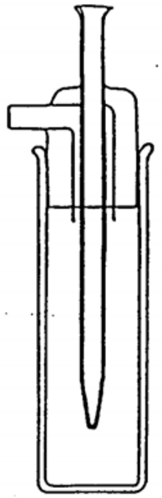
A - 100 mg sorbant stage
B - 50 mg sorbent stage

Carbon Adsorbent Tube

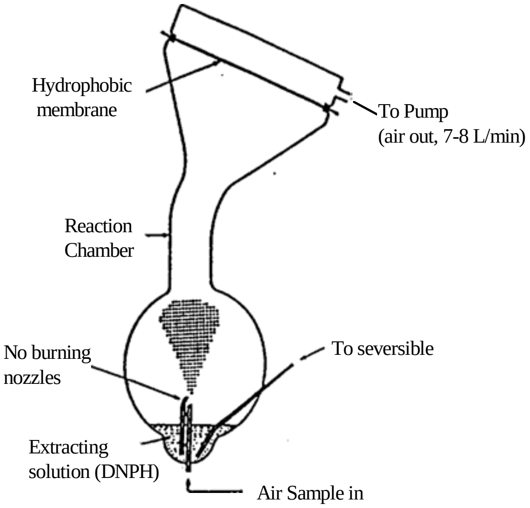
Figure 10-6A Air Component Concentration Samplers



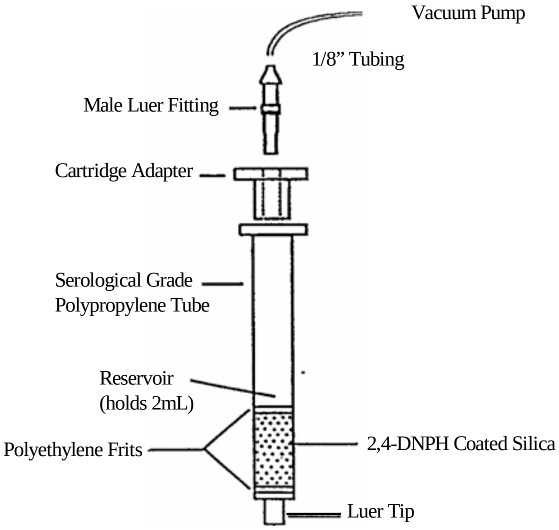
Bubbler



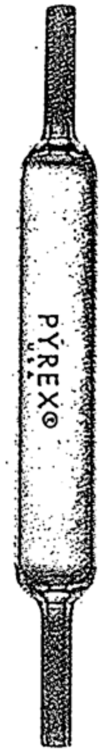
Impinger

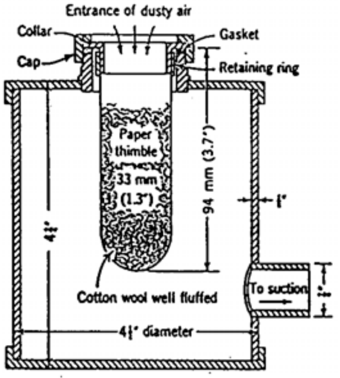


Reflux/Nebulization Concentrator



CNPH Cartridge for Monitoring Carbonyls





Thimble Filter

Denuder
(Empty Tube With Walls
Coated with Trapping Chemical)

Figure 10-6B Air Component Concentration Samplers

Disadvantages:

- 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

10.9.5.1.3 <u>Solid Sorbents</u>. Solid sorbents are the primary collection means used to remove 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 powder or foam. The organic chemicals are adsorbed from the air onto the surface of the adsorbent material. The laboratory removes the adsorbed chemicals by using solvents or heat.

Unfortunately, solid sorbents are not selective for particular chemicals. Since all organic 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, such as:

- **Organic Polymers**, including:
  - **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 Method 0030 &3.1.**
  - **Amberlite® XAD-2**. XAD-2, used in air sampling for polynuclear aromatic hydrocarbons, 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 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 remove semi-volatile chemicals such as pesticides, PCBs, and dioxins from large volumes of air, achieving a very low detection limit.

- **Inorganic Powders**, including **silica gel, Porasil® and 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 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® has been used to collect chlorinated pesticides and PCBs from air.

- **Carbon Adsorbents.** The carbon materials are much more adsorbent than are the organic polymers, and therefore allow efficient collection of highly volatile materials 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 materials such as benzene or toluene 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 from Rohm and Haas Chemical Company under the 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 microporous structure which leads to difficulty in recovering adsorbed materials. Therefore, this material is rarely used as a sampling media.

Table 10-4 Collection Methods for Toxic Organic Air Contaminants

Sampling Media	EPA Method
Solid adsorbent	
Tenax®	TO-1; 0030
Amberlite® XAD-2	TO-13; 0010; 0020
Thermosorb/N	TO-7
Carbon molecular sieve	TO-2
Silica gel (Porasil®; Florisil®)	none
Polyurethane Foam	TO-4; TO-9; TO-10; TO-13
Chemical Derivation	
Reactive coating on solid matrix	TO-11
Liquid impingers	TO-5; TO-6; TO-8
Cryogenic trapping	TO-3; TO-12
Nebulization/Reflux Concentration	Non Specified

- **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. 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 adsorbing liquid.

  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. Interest in determining carbonyl compounds (aldehydes and ketones) in ambient air has increased because these compounds are important in many photochemical reactions. They are sources of free radicals and precursors to oxidants such as ozone and peroxycarboxyclic nitric anhydrides (PANs). They are also sources of organic aerosols, and products of hydrocarbon oxidation. Additionally, carbonyls are produced in auto exhausts and are present in other naturally occurring and man-made processes.

  They are generally difficult to sample and analyze because their concentration is normally very low, and there are interferences from other compounds such as ozone. While the DNPH methodology can be used in an impinger set-up, it is not the most effective procedure. It suffers from many of the limitations of all impinger methods, and additionally is difficult to control for quantitative recovery of carbonyls due the competing processes of derivative formation and analyte volatility during the short solution-analyte contact time.

  Use of cartridges of solid sorbents coated with DNPH provide much higher collection efficiencies for carbonyls. A variety of sorbent media can be used including: silica gel, glass beads, glass fiber filter, XAD-2 resin, Florisil®, Chromosorb P®, Carbopak B®, and C18 coated silica. The solid sorbent method results in much better sensitivity by providing an in-situ analyte concentration in the sample. 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

- **Nebulization/Reflux Concentration:** This method is a modification of the bubbler/impinger methodology. In this case, instead of bubbling the air sample through the 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 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 10-5 EPA Standard Air Contaminant Sampling Methods for Toxic Organic Compounds

Method Number	Description	Types of Compounds Determined
TO-1	Tenax® GC adsorption and 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); 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 Gas Chromatography (GC) and High Performance Liquid Chromatography (HPLC) detection	Polynuclear Aromatic Hydrocarbons (PAHs)

Source: *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.* EPA/600/4-89/017. NTIS PB90-127374.

- **Cryogenic trapping.** The collection of organic chemicals from ambient air is an attractive alternative to adsorption or whole air collection. The disadvantages of various cryogens are listed in **Table 10-6**. A primary disadvantage of most cyrogenic methods is the tendency for water to condense with the sample.

  The design of a suitable sample trap is extremely important in cryogenic sampling. The residence time must be great enough to cool the air stream and condense the chemical of interest. The trap material must be able to withstand the wide temperature range involved in the sample process. The trap must also be appropriate for the recovery step, allowing efficient recovery without loss or contamination of the sample. For highly volatile chemicals, it may be necessary to include an adsorption medium such as silica gel to obtain satisfactory collection efficiency.

  A simplified cryogenic Pre-concentration, Direct Flame Ionization Detection (PDFID) method (Method TO-12) has been developed and recommended for use by state and local air pollution control agencies in developing their ozone control plans.

10.9.5.1.4 Mult-Functional Portable Commercial Samplers. Commercial samplers are available which combine capabilities for collection of multiple bag and sorbent tube samples. Some also have an easily portable weather station capable of monitoring wind speed, wind direction, temperature, relative humidity, barometric pressure, precipitation, and solar radiation.

Table 10-6 Cryogens Used in Air Sampling

Cryogen	Disadvantages
Liquid nitrogen	Not acceptable because large quantities of air will condense
Liquid oxygen	Satisfactory, except fire hazard
Liquid argon	Satisfactory, except for relatively high cost
Ice water	Not cold enough for collecting organics in ambient air except for relatively nonvolatile compounds
Dry ice/ solvent	Should be employed with caution because of the high probability of contaminating the sample with relatively large quantities of the solvent

10.10 SOLID 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 chemicals 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 10-7** for 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, impinger, etc.) downstream from the particulate capturing device. Analysis of both collection media should be necessary to determine the total amount of chemical present in the ambient air, but the results may not reflect the actual phase distribution of the chemical in the air.

10.10.1 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:

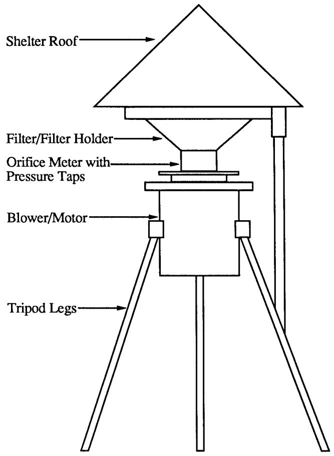


Figure 10-7 Particulate Sampler

- Cellulose fiber (e.g., Whatman No. 41 filter paper)
- Glass or quartz fiber - withstand higher temperatures, 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

Table 10-7 Liquid Absorbers Commonly Used in Gas or Vapor Sampling

Absorbing Liquid	Gas/Vapor Absorbed
0.1N H_2SO_4	Bases and Amines
0.1N NaOH	Acids and Phenol
0.1N HCl	Nickel Carbonyl
Alkaline $CdSO_4$ ($CdSO_4$ NaOH)	Hydrogen Sulfide
Methylene Blue	Hydrogen Sulfide
1% KI in 0.1N NaOH	Ozone
Nitro Reagent (4-nitropyridyl propylamine in toluene)	Di-isocyanates
0.3N H_2O_2	Sulfur Dioxide
0.1% Aniline	Phosgene
1% $NaHSO_2$	Formaldehyde
Distilled Water	Acids and Bases

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.

The EPA has specified the use of fiberglass filters for the collection of total suspended particulates in the following standard reference methods:

- **Total Suspended Particulates in Air:** The standard EPA reference method is specified in **Appendix B to 40 CFR Part 50**. This procedure requires that air be drawn for 24 hours through a fiberglass filter with a flow rate of 1.1 to 1.7 cubic meters per minute. The filter must have an area of 63 square inches and must be 99% efficient in removing aerosol droplets 0.3 micrometers in diameter. The filter is weighed before and after use to determine the net weight gain. The approximate concentration range of this method is 2 to 750 micrograms per standard cubic meter.
- **Lead in Total Suspended Particulates:** The standard EPA reference method is specified in **Appendix G of 40 CFR Part 50**. This method collects particulates from air using the procedure described in **Appendix B of 40 CFR Part 50** for total particulates. Acid is used to extract the lead from the particulates on the filter and the concentration of lead in the acid is measured in the laboratory. This method has a range of 0.07 to 7.5 micrograms lead per cubic meter.

10.10.2 Centrifugal Collection and Impaction. EPA ambient air standards for particulates are based on the concentration of particles having an aerodynamic diameter of less than 10 micrometers. Particles larger than this are not trapped in the lungs and are, therefore, less hazardous to heath. 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.

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 the aerosols are deposited. The solid surfaces may be a baffled chamber, slotted plate, channels, or other obstructive arrangement. The primary use is separation of mists from gases. **Table 10-7** lists liquid absorbers commonly used in gas or vapor sampling.

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.

PM10/PM2.5 Separators. These devices separate particles in broad cuts of <10 microns and/or <2.5 microns. They are frequently used preceding one or more filter packs.

In **Appendix J of 40 CFR Part 50**, the EPA specifies the use of centrifugal collectors for the determination of PM_{10} particulate matter in the atmosphere. PM_{10} is defined as particles having an aerodynamic diameter less than or equal to 10 micrometers. Samples are collected for a period of 24 hours by drawing ambient air into a specially shaped inlet where the suspended particulate is inertially separated into one or more size fractions within the PM_{10} size range. Each fraction is then collected on a separate fiberglass filter weighed before and after use to determine the net weight gain due to collected PM_{10}.

10.11 OTHER DEVICES USED TO COLLECT AEROSOLS.

Wet Collectors. These use a finely dispersed liquid to increase size of aerosol particles.

Electrostatic separators. These use high voltage to ionize aerosols and then coalesce and collect them. For the method to be effective, the aerosol must have a suitable ionization potential. The method works well for acid mists, but poorly for compounds such as carbon black.

10.12 SPECIFIC APPLICATION OF GAS AND AEROSOL TECHNOLOGIES - DRY DEPOSITION SAMPLING.

The concentration and rate of deposition of gases and particulates from the atmosphere are a matter of extreme concern because the concentration of these materials in the atmosphere significantly affects the dispersion or disposition of other atmospheric pollutants. In recent years, the EPA, National Park Service, and some states have set up multiyear monitoring programs to build databases to assess dry deposition fluxes. Monitoring of this type is most frequently done at a number of sites simultaneously. Meteorological parameters and topological features are extremely important considerations for correct interpretation of dry deposition data.

A number of types of collection devices are used to collect dry deposition samples, often in sampling trains, where the air passes from one collector to the next.

Cellulose, Glass Fiber, and Polymeric Filters. Filters of this type can be used with or without chemical impregnation to selectively remove particulates of a certain size range or specific analytes. They can be used individually, or in combination in two or three stage filter packs for the following separations:

- Teflon® membrane filters which separate particles under 10 microns (PM10) or 2.5 microns (PM2.5)
- Nylon filters to remove NO_2 gas
- Cellulose filters impregnated with 15 weight percent potassium carbonate and 5% glycerol in water for SO_2
- Cellulose filters impregnated with 25 % citric acid and 5% glycerol in water for NH_3
- Cellulose filters impregnated with 25% TEA , 4% ethylene glycol, 25% acetone in 46% water for NO_2

Frequently, several types of filters with different chemical selectivities may be combined in a filter pack. Filter packs consist of a holder constructed of inert material which will not absorb acidic gases (preferably Teflon®), which has a tapered end to fit the sampling collector. In the bottom of the holder is a support structure topped by a grid screen which supports the first stage filter disk. This is topped by a Viton® o-ring, another support grid screen and the second stage filter disk. After the desired number of stages are assembled, a retaining device fits into the top of the filter pack. Filter packs may be used either in-line with the sampler, or with one side open. Generally, a better distribution of collection is obtained when one face of the sampler is open.

Great care must be taken in cleaning, preparation, handling, assembly, and field procedures with filter packs. Contamination is a significant concern. In choosing filter materials, the following factors should be considered:

- Physical and mechanical durability of material
- Chemical stability
- Sampling efficiency for analytes of interest
- Resistance to flow (back pressure level)
- Adequacy of loading capacity
- Ease of obtaining pure blanks
- Tendency to produce artifacts
- Compatibility to sampling and analytical methods
- Cost
- Commercial availability or ease of preparation

Denuders. Denuders are generally glass tubes which are coated on the inside with a thin film of adsorbent chemical specific for collection of certain gaseous analytes. Sometimes, the tube may also be packed with beads which are also coated.

10.13 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, 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.

Table 10-8 provides guidance on QA and QC requirements for stationary source testing. **Table 10-9** provides guidance on report formats for stationary source testing.

10.13.1 <u>Special Problems in Stack Vent and Duct Sampling</u>. 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 that in the source in order to minimize the turbulence caused by changes in gas flow at the tip of the probe. **When the gas flow rate and flow direction through the nozzle tip is equal to the flow rate of gas passing through the source, the sampling conditions are 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.

Figure 10-8 shows a typical sampling apparatus for stack gases.

10.13.2 <u>Specified Procedures</u>. In order to ensure the accuracy of reported stack, vent or duct sampling data, the EPA specifies the sampling procedures in great detail. **Table 10-5** lists the EPA standard procedures and the chemicals measured by each. 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.

Table 10-8 QA and QC General Checklist for HAP Emission Testing

Category	Sampling	Analysis	Reporting
Technical Responsibility	Yes	Yes	Yes
Quality Assurance Responsibility	Yes	Yes	Yes
Data Quality Objectives	Yes	Yes	Yes
Quality Control Procedures			
Equipment Calibration	Yes	Yes	No
Sample Custody Chain	Yes	Yes	No
Measurement Traceability	Yes	Yes	No
Blanks	Yes	Yes	No
Control Checks		Yes	No
Calibration Standards		Yes	No
Storage Time	Yes	Yes	No
Sample Spikes	Optional	Yes	No
Internal Standards		Optional	No
Surrogates		Optional	No
Replicates	Optional	Yes	No
Linearity		Yes	No
Leak Testing	Yes	Yes	No
Data Review	Yes	Yes	Yes
Quality Assurance Procedures			
System Audits	Optional	Optional	Optional
Performance Audits	Optional	Yes	No
Written SOP	Yes	Yes	Yes
Documentation	Yes	Yes	Yes

Source: Principles of Environmental Sampling, L. H. Keith, Ed., ACS Professional Reference books, ACS, 1988

Table 10-9 Suggested Source Testing Report Format

Section	Topics
Cover	1. Plant name and location 2. Source(s) sampled 3. Testing company or agency, and name and address
Certification	1. Certification by team leader or technical management 2. Certification by technical reviewer (e.g., P.E.)
Introduction	1. Test purpose 2. Test location(s) and type of process(es) 3. Test dates 4. Pollutants tested 5. Observer's name and affiliations (industry and agency) 6. Any other important background information
Summary of results	1. Emission results, with tables and figures (wherever possible, these should contain coefficients of variation, standard deviations, and error bars) 2. Process data as related to determination of compliance 3. Allowable emissions 4. Description of collected samples 5. Discussion of errors, both real and apparent
Source Operation	1. Description of process and control devices 2. Process and control equipment flow diagram 3. Process data and results, with example calculations 4. Representativeness of raw materials, products and operation 5. Any specially required operation demonstrated
Sampling and Analysis Procedures	1. Sampling port location(s) and dimensioned cross section 2. Sampling point description, including labeling system 3. Sampling train(s) description 4. Brief discussion of sampling procedures, with deviations from standard methods 5. Brief discussion of analytical procedures, with deviations from standard methods
QA and QC Procedures	1. Data quality objectives 2. QC results for precision and accuracy 3. Comparison of QC results with goals 4. Discussion of QA and QC documentation provided in the appendix to establish traceability to primary standards
Appendix	1. Complete results, including sample calculations 2. Raw field data (copies of originals) 3. Laboratory reports, including chain of custody 4. Raw production data signed by plant official 5. Calibration procedures and results 6. Test logs 7. Project participants and titles 8. Related correspondence 9. Standard procedures (excluding 40 CFR 60 methods)

Source: Principles of Environmental Sampling, L. H. Keith, Ed., ACS Professional Reference books, ACS, 1988

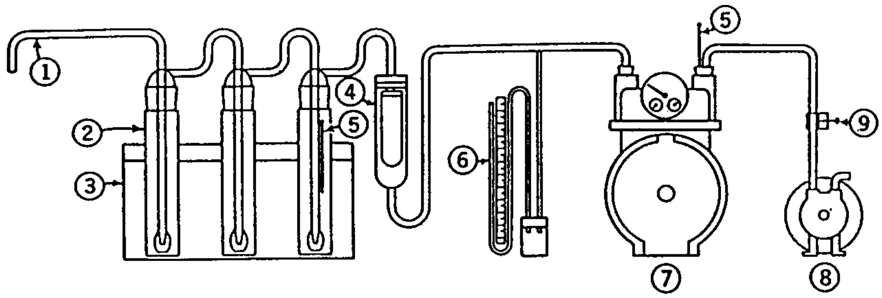
10.13.2.1 Method 0010 - Modified Method 5 Sampling Train. Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. Principal components of the train include a high efficiency glass or quartz fiber filter 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.

10.13.2.2 Method 0020 - Source Assessment Sampling System (SASS). Particulate and semi-volatile organic materials are withdrawn 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.

10.13.2.3 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 Volatile Organic Sampling Train. 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.

10.14 HAZARDS 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. Refer to **Chapter 3, Section 3.3.3** and **Appendix D** for specific hazard identification and appropriate reference for detailed control measures.

10.14.1 Environmental. 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.



Sampling apparatus for stack gases. (1) Sample probe. (2) Impinger (dust concentration sampler). (3) Ice bath container. (4) Dry filter. (5) Thermometer. (6) Mercury manometer. (7) Sprague dry gas meter (Zephyr No. 1A). (8) Vacuum pump. (9) Hose clamp to control gas flow rate. (*Courtesy Los Angeles Air Pollution Control District.*)

Figure 10-8 Sampling Apparatus for Stack Gases

Source: Faith, W. L. and Atkinson, A. A, Jr.

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

10.14.2 Stack, Vent, and Duct Sampling Safety.

WARNING

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, including criteria for use of PPE, such as fall protection lanyards and heat resistant (Nomex®) coveralls and gloves.

10.14.3 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.

10.15 PREPARATION FOR AIR SAMPLING.

10.15.1 Role of the Sampler. The role of sampling personnel is critical to the over all conduct of an air sampling program. The role of sampling personnel is to:

- Prepare or review Field Sampling Plan (FSP)
- 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

10.15.2 Guidelines for Air Sampling. The FSP 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 FSP 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 shelf life has not expired
    - 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 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. Double check that a sufficient number of sample canisters and other consumable equipment is available to cover sampling requirements.
11. Obtain a **Field Log Book/Field Notes**. Keep air monitoring field notes neatly and legibly in a legally defensible form. When sampling begins, record in the **Field Log Book/Field Notes**, the start time, flow rate, weather conditions (ambient temperature, barometric pressure, humidity, etc.), locations of sampling equipment, and other pertinent information. 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 FSP 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, vermiculite, or other cushioning material to prevent breakage during shipment to and from the site.

10.16 <u>GENERAL AIR SAMPLING PROCEDURES</u>. 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 **FSP** specifies a standard EPA procedure, a complete copy of the procedure should be obtained and reviewed to ensure necessary equipment is available.

NOTE:

Specified steps of the EPA procedure should be followed as exactly as possible. **Any deviations from the procedure should be noted in detail in the Field Log Book/Field Notes.**

The following sections describe the use of real-time air monitoring equipment and present introductory information on the use of cartridge samplers. This information is included to assist in understanding the requirements of the detailed EPA procedures. **In all cases, the procedures specified by the FSP must be used.**

10.16.1 <u>Real-Time Monitoring</u>. Real-time air monitoring is mostly used for workplace and emergency response situations because detection limits are generally in the range of acute health hazards. The calibration procedures, required maintenance, and limitations may differ among types, brand names, and models of field instruments. Refer to **Section 10.8** concerning safety precautions with field instruments.

NOTE:

The use of real-time air monitoring equipment is not covered by EPA standard methods, therefore, it is important that the calibration and use of this equipment be thoroughly documented in the **Field Log Book/Field Notes**. Information to be recorded depends on the type of sampling being performed. Not all parameters are applicable for all types of sampling (e.g. ambient air, indoor air, and stack sampling have different data requirements), the following data categories are intended as guidelines.

The **Field Log Book/Field Notes** is the primary record of the air monitoring data collected by real time air monitoring equipment. The book should contain the following information:

Calibration. Record the following information:

Time of calibration
Name and signature of the person doing the calibration
Equipment type, manufacturer, model, and serial number
Calibration standard (e.g., cylinder manufacturer, product number, serial number)
eV rating of bulb type (PID detectors)
All maintenance done on or repairs made to the equipment, including disassembly for cleaning, reason for repair, and description of work performed
All adjustments made to the equipment for proper calibration

Field readings. Include the following information:

NOTE:

For equipment that has data-logging capability, include this measurement each time the event key is pushed and for each significant change in location or change in conditions that are noted.

Time of measurement
Instrument response (dial or display reading)
Event number (if used)
Location of measurement - include site sketch and indicate location with reference to fixed points
Weather information, including wind speed, temperature, precipitation, etc.
Description of circumstances that could affect the reading (e.g., downwind of heavy truck traffic, probe one foot above spill, downwind of momentarily opaque stack release, etc.)
Names of workers in the immediate area when the sample was collected
Any fault indication on the instrument display, including time of fault, and actions taken

- **Data log.** Some instruments store periodic measurements in an internal computer memory. The memory includes the instrument reading, the time, and the status of an event key. The stored information can be downloaded to a computer at the end of the day and then printed out. A note should be made in the Field Log Book/Field Notes if data is downloaded and printed out. The printout should then be annotated with data from the **Field Log Book/Field Notes** for each significant event (location change, reason for pressing event key, etc.) and signed by the person who performed the air monitoring. The original of the annotated printout should be sent to the Program Manager or designee, with a copy being placed in the **Field Log Book/Field Notes**.

10.16.2 Real-Time Monitoring for Volatile Organic Chemicals (VOCs). Real time air monitoring for VOCs is performed to identify acutely hazardous conditions. The data is used to determine whether respirators must be worn in the area (and if so, what kind), and to establish the limits of the Exclusion Zone where respirators are required and to identify if process equipment or waste containers need control devices. Real-time monitoring can also be performed at site boundaries during emergency conditions to determine whether air leaving the site is hazardous. Real-time VOC monitoring is also done to defend against litigation claiming personal injury as the result of negligence on the part of the supervisors. Therefore, all monitoring must be done with attention to detail and with complete documentation.

10.16.2.1 PID Operation. Operation of Photo Ionization Detectors varies depending on the manufacturer and is often complex. Manufacturers may offer training sessions on their units. Regulations may dictate instrumentation specifications as well. See **Chapter 14** for some examples of specific SOPs.

In general, the steps required to operate a PID are as follows:

1. **PREPARATION.**
    - 1.1 Ensure the instrument is adequately charged.
    - 1.2 Ensure the proper lamp is installed for detecting the chemicals of interest.
    - 1.3 Turn on the unit and allow it to warm up.
    - 1.4 Check for obvious operational problems and troubleshoot accordingly.
    - 1.5 Check instrument settings and alarms and adjust accordingly.
    - 1.6 Check data logging functions for proper settings and operation.
    - 1.7 Calibrate the instrument using the appropriate calibration gas. Note calibration in the Field Log Book/Field Notes.
    - 1.8 Obtain required spare parts and tools that may be required for field maintenance and troubleshooting.

2. **AIR SAMPLE MEASUREMENT**
    - 2.1 Recheck calibration.
    - 2.2 Refer to paragraph 10.16.1 for required Field Log Book/Field Notes entries.

      For monitoring headspace (area close to liquid surfaces), take care always not to draw any liquid into the instrument.

    - 2.3 Place the probe in the area of interest and monitor readings. Allow adequate response time (about two times the instrument's rated response time).

10.16.2.2 FID Operation. Like the PID, operation of Flame Ionization Detectors vary depending on the manufacturer and is often complex. Manufacturers may offer training sessions on their units. Regulations may dictate instrumentation specifications as well. See **Chapter 14** for some examples of specific SOPs.

CAUTION

In general, the steps required to operate a FID are as follows:

1. **PREPARATION.**
   - 1.1 Ensure the instrument is adequately charged.
   - 1.2 Ensure the Hydrogen gas cylinder is installed and contains an adequate quantity for sampling.
   - 1.3 Turn on the unit and allow it to warm up.
   - 1.4 Check for obvious operational problems and troubleshoot accordingly.
   - 1.5 Check instrument settings and alarms and adjust accordingly.
   - 1.6 Check data logging functions for proper settings and operation.
   - 1.7 Calibrate the instrument using the appropriate calibration gas. Note calibration in the Field Log Book/Field Notes.
   - 1.8 Obtain required spare parts and tools that may be required for field maintenance and troubleshooting.

2. **AIR SAMPLE MEASUREMENT.**
   - 2.1 Recheck calibration.
   - 2.2 Refer to paragraph 10.16.1 for required Field Log Book/Field Notes entries.

CAUTION

For monitoring headspace (area close to liquid surfaces), take care not to draw any liquid into the instrument.

   - 2.3 Place the probe in the area of interest and monitor readings. Allow adequate response time (about two times the instrument's rated response time).

10.16.2.3 Infrared Absorbance Spectrometer Operation. Infrared absorbance detectors must be calibrated to the specific chemical of interest. Calibration procedures depend on the instrument. In general, a calibration gas mixture is needed for each chemical to be measured. The absorbance chamber must be flushed with clean air after each measurement to reset the zero point.

Calibration and maintenance procedures are dependent on the particular manufacturer and model of instrument used. In general, the following items are required:

- Spare battery
- Battery charger
- Spare particulate filter
- Zero gas filter (filters all VOCs out of air to reset the zero point)
- Calibration gas for each chemical to be measured

10.16.3 Hydrogen Sulfide, Carbon Monoxide, and Flammable Vapors Meters Operation. Combination meters are available that incorporate sensors for flammable vapors and oxygen levels. Some of these instruments also have additional sensors for hydrogen sulfide, carbon monoxide, and/or other specific chemicals.

CAUTION

Do not calibrate with air from inside a building. Outside air can be used to set the zero adjustment for flammable vapors and to adjust the oxygen calibration to **21%**. Calibrate the explosimeter and other gas detectors at least once each day using gas from calibration cylinders. Follow the manufacturer's instructions for the particular instrument for calibration and maintenance.

CAUTION

The sensor elements can be damaged by exposure to certain conditions, and will wear out. Therefore, it is recommended that additional sensor elements be available for use if the instrument loses sensitivity or cannot be calibrated.

The following parts should be available when using these combination field instruments:

- Extra particulate filters
- Extra dry cell batteries
- Calibration gas cylinders
- Plastic bags for calibration procedure

10.16.4 Aerosol Meter Operations. Aerosol monitoring meters are generally calibrated in the factory once each year. Field use requires checking battery status, and ensuring there is an indicator response when the unit is exposed to an aerosol such as smoke.

10.16.5 Mercury Vapor Meter Operation. Mercury vapor monitoring instruments cannot be calibrated in the field, but a number of routine maintenance procedures are required to ensure satisfactory operation:

Battery - A backup battery should be available to replace the battery in the unit when it becomes low. Rechargeable batteries should be fully charged when installed and allowed to discharge at least 95% before being recharged.

Regenerating gold film: The reheating cycle that removes mercury from the gold film sensor requires about 15 minutes. This procedure generally requires that a special filter be attached to the sampling port, and that the instrument be attached to line power. The sensor should be regenerated:

- Whenever the meter reading indicates that the sensor is saturated
- At the end of each day's use
- To reinitialize the system after long term storage

Remove system contamination: All components in the flow system are subject to contamination from high mercury concentrations in the air. To determine whether the instrument is contaminated, attach the filter to the inlet port and press the sample button three times. If the instrument reading is not essentially zero, replace the filter with a new filter and press the sample button three times. If the reading is still above zero, change the internal filter and tubing. If the reading is still significantly above zero with the filter in place, return the unit to the manufacturer for service.

10.16.6 <u>QC Sample Preparation of Passive Samplers</u>. Quality control samples can be generated by exposing one sampler from each batch to a calibration gas for the normal sampling period. To prepare a QC sample:

- Open the sampler and place it in a plastic bag.
- Empty the bag using a vacuum (or air sampling) pump.
- Fill the bag with calibration gas from a cylinder.
- Enter into the **Field Log Book/Field Notes**:
  - Sampler type, manufacturer, lot number
  - Date, time, person preparing the sample
  - Calibration cylinder manufacturer, type, lot number, label information on the concentration of the chemical(s) of interest
- Leave the badge in the bag for the normal sampling period, then remove and handle as a normal sample:
  - Assign sample number
  - Enter on **Chain-of-Custody** Record
  - Send to lab for analysis

In addition, one blank sample should be submitted to the lab with each quality control sample. To prepare a blank sample, perform all of the steps associated with the normal use of the sample, but do not expose it to contaminated air.

10.16.7 <u>Sample Collection Using Active Samplers</u>. Active samplers pass a measured quantity of ambient air through a collection media such as a solid adsorbent, liquid impinger or particulate filter that is chosen for its ability to retain the chemical(s) of interest. For each sample, follow the standard EPA (NIOSH) sampling method procedures.

10.16.7.1 <u>Measurement of Air Volume Sampled</u>. To obtain the proper results, the total volume of air sampled must be known. The flow rate of air through the sampling media should 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. If the sampling pump is connected to line power and does not contain a timer, an inexpensive 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:

Total Volume = Average Flow Rate X Time

10.16.7.2 <u>Low Flow Rate Samples</u>. The most common type of air flow measuring device for low flow rate air samples is the soap film calibrator as

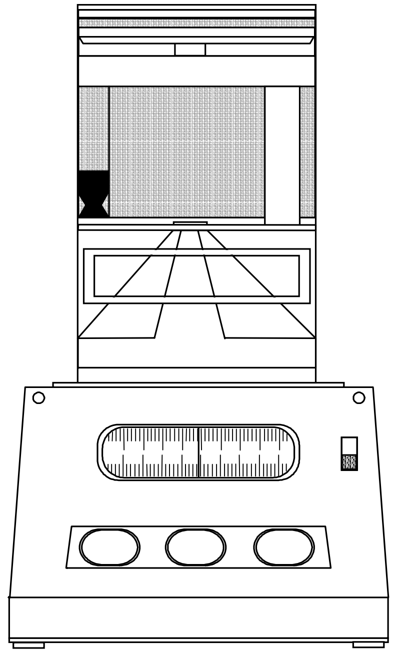


Figure 10-9 Soap Bubble Air Calibrator

shown in **Figure 10-9**. 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.

10.16.7.3 **<u>High Flow Rate Samples</u>.** 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 is calculated by multiplying the average flow rate by the sampling time.

10.16.7.4 **<u>High Flow Rate Sampling for Semi-Volatile Organic Chemicals</u>.** The type of air sampler used should depend on the monitored contaminant. Usually a two stage sampling system is used. Air first passes through a borosilicate fiberglass filter which traps particulates and then passes through a cartridge containing **polyurethane foam (PUF) filters** which trap semi-volatiles that are in the air or that evaporate off the particulates.

At the completion of sampling, the sampling media must be carefully packed. The fiberglass particulate filters should only be handled with stainless steel tweezers and non-powdered surgical gloves. The filter should carefully be folded with the dirty side "in" so that it can fit into a sample jar. The PUF filters should also be handled carefully. If there is more than one in the cartridge, they should be packed in the sample jar in the same order they were stacked in the cartridge. Some PUF cartridges are designed so that the entire cartridge can be sealed and shipped for analysis.

If the particulate filter and the PUF filters are placed in separate jars, each jar should be given a separate sample number and the **Chain-of-Custody** Record should note which sample containers hold parts of the same sample. If the entire cartridge is sent, it should be wrapped in foil, with the top part of the sample label attached to the cartridge.

10.16.8 **<u>Particulates Sampling using Fiberglass Filters</u>.** **Borosilicate fiberglass filters** are used to trap particulates. Accurate analytical results depend on sufficient drying and accurate weighing of the filters. Filters must be dried for 24 hours prior to use, then weighed to determine the clean weight. An analytical balance accurate to 0.001 gram must be used.

Filters should be handled with clean stainless steel tweezers and non-powdered gloves to avoid contamination and particulate loss. After the filters are used, they must again be dried for 24 hours and then weighed to determine the dirty weight. The difference between the dirty weight and the clean weight is the total particulate weight. The concentration of Total Suspended Particulates (TSP) is determined by dividing the total particulate weight by the volume of air sampled.

$$TSP = \frac{Final\ Weight\ -\ Initial\ Weight}{Total\ Air\ Volume}$$

Where:

TSP = Concentration of suspended particulates per volume of air

If the filters are shipped to a laboratory for weighing and/or analysis, they must be carefully packaged to avoid the loss of particulates during shipment. The preferred sample container for a particulate filter is a petri dish (shallow glass dish with a glass cover) having a diameter slightly larger than the filter. If glass containers are not available, the filter should be placed on a piece of aluminum foil with the foil folded around the filter. The filter must not be folded. The top part of the three-part label is attached to the foil enclosing the samples. The filter and foil are then placed in a plastic bag and sealed. The volume of air sampled should be included on the **Chain-of-Custody (COC)** Record.

10.17 **<u>EXAMPLES OF SPECIFIC AIR SAMPLING PROCEDURES</u>.** Air sampling procedures depend highly on the instruments used and, therefore, will require specific SOPs. **Chapter 14** contains an example of an Standard Operating Procedure for PID calibration and maintenance.

10.18 **<u>REGULATORY REQUIREMENTS</u>.** The EPA regulates air quality under numerous laws and guidelines including, but not limited to, those outlined below.

10.18.1 **<u>Clean Air Act (CAA)</u>.** The CAA requires

EPA to establish national ambient air and emission air standards. EPA regulations related to the CAA are contained in Title **40 CFR** Parts **50 through 87**.

10.18.1.1 National Ambient Air Quality Standards (NAAQS). 40 CFR Part 50 establishes the primary and secondary NAAQS. NAAQS are established for the following:

- Sulfur oxides - SO_2
- Carbon monoxide - CO
- Particulate Matter - PM_{10}
- Ozone -0_3
- Nitrogen dioxide -NO_2
- Lead or lead compounds

The 1990 CAA Amendments included new requirements for non-attainment areas, mobile sources (including possible reformulation of gasoline), hazardous air pollutants (initial list contains 189 pollutants), acid rain control, new permits, and stratospheric ozone protection.

10.18.1.2 National Emission Standards for Hazardous Air Pollutants (NESHAPS). The EPA has established emission standards for hazardous air pollutants for a number of industries. NESHAPS promulgated prior to the clean air act amendments of 1990 (CAA90) are located in 40 CFR part 63. The following sub-paragraphs summarize requirements of particular significance to Navy operations.

10.18.1.2.1 Beryllium Machine Shops. As specified in **40 CFR 61.30**, any shop machining beryllium, beryllium oxides, or any alloy containing over 5% beryllium is required to perform stack sampling to ensure that total emissions of beryllium do not exceed 10 grams over a 24 hour period. **Stack sampling procedures are specified as Methods 103 and 104 in Appendix B of 40 CFR 61**. Alternatively, the owner may request approval from the EPA to demonstrate that the concentration of beryllium in the ambient air around the source does not exceed 0.01 $\mu g/m^3$, averaged over a 30 day period.

10.18.1.2.2 Mercury in Sewage Sludge. As specified in **40 CFR 61.50**, mercury emissions from incineration or drying of waste water treatment sludge must not exceed 3,200 grams per 24 hour period. The stack sampling procedure is specified as **Method 101A in Appendix B of 40 CFR 61**. Alternatively, the owner may sample the sludge using the procedure specified as **Method 105 in 40 CFR 61 Appendix B**.

10.18.1.2.3 Fugitive Emissions of Benzene and Vinyl Chloride. Fugitive emissions are sources of air pollutants other than stacks and vents. As specified in **40 CFR 61.240**, process equipment containing or contacting a gas or liquid of more than 10% benzene and/or vinyl chloride must be checked periodically for leaks. Requirements apply to pumps, compressors, pressure relief devices, sampling connection systems, open-ended valves or lines, valves, flanges and other connectors, product accumulator vessels, and control devices/systems. Checks must quantify leaks by using a real-time air monitoring instrument sensitive to the chemical.

10.18.1.2.4 Aerospace Manufacturing and Rework Facilities. Regulation of major sources of Hazardous Air Pollutants (HAPs) associated with aerospace manufacturing and rework facilities are specified in 40 CFR 63 Subpart GG. These regulations affect facilities that are engaged, either in part or in whole, in the manufacture or rework of commercial, civil, or military aerospace vehicles or components that are major sources of HAPs. Affected operations include cleaning, topcoats and primers, depainting, and chemical milling maskants. Generally, these regulations affect only parts and assemblies of aerospace vehicles that are critical to structural integrity or flight performance, with the exception that cleaning requirements apply to spray guns, etc. Compliance can be achieved using compliant coatings and/or control devices.

Exemptions:

- Facilities that depaint less than six (6) aircraft per year are exempt from the depainting requirements. Nonoperational aircraft and radomes and other normally removed parts are also exempt from the depainting requirements. The rule also exempts the use of up to 50 gallons per military aircraft (26 gallons per commercial aircraft) of organic HAP containing strippers for decal removal and spot stripping.
- Sections 63.741(f), 63.741(g), 63.744(e), 63.745(f)(3), and 63.745(g)(4) contain numerous operation-specific exemptions and exceptions to the rule.

10.18.1.2.5 Chromium Electroplating and Anodizing Tanks. Major source and area sources of HAPs in chromium electroplating and anodizing are regulated under 40 CFR 63 Subpart N. These regulations affect all hard chromium electroplating, decorative, chromium electroplating, and chromium anodizing tanks. Hard chromium electroplating tanks must comply with an emission limit. Decorative chromium electroplating and anodizing sources must comply with either a surface tension limit or an emission limit.

10.18.1.2.6 Halogenated Solvent Cleaning. 40 CFR 63 Subpart T regulates major sources and area sources of HAPs associated with halogenated solvent cleaning.

All batch vapor, in-line vapor, in-line cold, and batch cold solvent machines that use any solvent containing methylene chloride, perchloroethylene, trichloroethylene, 1,1,1-trichloroethane, carbon tetrachloride, or chloroform -- or combination of these halogenated solvents -- in a total concentration greater than 5% by weight, as a cleaning or drying agent are regulated. Requirements vary depending on the type of equipment used and whether the source is existing or new. Compliance options include various combinations of design and control device requirements, alternating idling mode emission limits, or alternative emission limits.

10.18.1.2.7 <u>Perchloroethylene (PCE) Dry Cleaning</u>. Perchloroethylene dry cleaning major sources and area source HAPs are regulated under 40 CFR 63 Subpart M. Most new and existing PCE dry cleaning operations are affected, however, coin-operated (customer-operated) machines are exempt. The rule contains emission control and work practice requirements. Requirements vary depending upon the annual PCE consumption of the facility. Most facilities are achieving compliance by purchasing new ventless dry-to-dry machines.

10.18.1.2.8 <u>Shipbuilding and Ship Repair Facilities</u>. Major sources of HAPs associated with shipbuilding and ship repair facilities are regulated under 40 CFR 63 Subpart II. Shipbuilding and ship repair operations at any facility that are major sources of HAPs are affected. Compliance can be achieved using compliant coatings and/or control devices.

Exemptions:

- Coatings used in volumes of less than 200 liters (52.8 gallons) per year can be exempted provided the total volume of coating exempted does not exceed 1,000 liters (264 gallons) per year at any facility. These exempt coatings shall be clearly labeled as "low-usage exempt", and the volume of each such coating applied shall be maintained in the facility's records.
- Coatings applied with hand-held, non-refillable, aerosol containers can be exempted.
- Unsaturated polyester resin (i.e., fiberglass lay-up) coatings can be exempted. However, coatings applied to suitably prepared fiberglass surfaces for protective or decorative purposes are subject to this subpart.

10.18.1.2.9 <u>Wood Furniture Manufacturing Operations</u>. Major sources of HAPs used in wood furniture manufacturing operations are regulated under 40 CFR 63 Subpart JJ. New and existing wood furniture manufacturing operations located at plant sites that are major HAP sources are affected. This rule does not apply to refinishing operations, only original furniture manufacture. The final rule exempts "incidental wood furniture manufacturers" from all requirements except that they must keep records of coating purchases and usage. An *incidental wood furniture manufacturer* is a major source that uses no more than 100 gallons per month of finishing material or adhesives in the manufacture of wood furniture or wood furniture components.

10.18.1.2.10 <u>Solid Wood Combustion Standards</u>. The CAA90 requires EPA to regulate various types of solid waste combustors for HAPs. Combustors targeted for regulation include municipal waste, medical waste, residential waste, agricultural waste, wood waste, construction and demolition waste, crematories, and contaminated soil treatment facilities.

10.18.1.2.11 <u>Municipal Waste Combustors (MWC)</u>. EPA promulgated new source performance standards (NSPS) in 40 CFR 60 Subparts Ea and Eb and existing sources emission guidelines in Subpart Cb for municipal waste combustors capable of combusting greater than 35 megagrams (Mg) per day. Requirements include emission standards for criteria and HAPs, good combustion practices, and monitoring.

Exemptions:

- **MWC Plants Permitted to Combust Less Than 10 Mg/day.** Any medical, industrial manufacturing, or other type of waste combustor plant capable of combusting more than 35 Mg/day of Municipal Solid Waste (MSW) but actually combusting less than 10 Mg/day of MSW (plant-wide) is not subject to this rule, provided it submits an initial report containing a copy of the plant's federally enforceable permit limiting the amount of MSW that may be combusted by the plant to less than 10 Mg/day and keeps records on the daily weight of MSW fired.
- **Cofired Combustors.** Cofired combustors (that combust less than 30 percent MSW) located at a plant with an aggregate plant combustion capacity greater than 35 Mg/day are exempt from the requirements of the guidelines provided they submit a notification of exemption and keep records of the weight of MSW combusted on a calendar quarter basis.
- **Used Oil Combustors.** The definition of MSW excludes used oil. Although used oil combustors are not affected by these rules, MWCs that combust used oil in addition to MSW will be affected unless they qualify for either the "cofired combustor" or "10 Mg/day plant" exemption.

10.18.2 <u>State Air Quality Programs</u>. 40 CFR Part 51 contains the requirement for each state to prepare a **State Implementation Plan (SIP)** for EPA approval that describes how it will achieve the established national air quality goals. The states are responsible for the execution of these plans after approval. They issue air quality permits, enforce the requirements established in their plans, and conduct reviews of all new major sources of air pollutants. The EPA requirements for state air quality operating permit programs are specified in **40 CFR 70**.

State Implementation Plans (SIPs) implement pollution control programs such as **New Source Performance Standards (NSPS), New Source Review (NSR),** and **National Emission Standards for Hazardous Air Pollutants (NESHAPs)** at the state and local level.

10.18.3 **<u>Acid Rain Permits</u>.** EPA regulations **40 CFR 72** require states to establish an Operating Permit Program for fossil fueled electric generating power plants to protect the environment from acid rain. Associated continuous emission monitoring requirements for CO_2, NO_x, and SO_2 are specified in **40 CFR 75**.

WARNING

10.18.4 **<u>Emission Limitations - New Stationary Sources</u>**. The EPA has established specific limits on stack emissions for a number of industries. These performance standards are listed in **40 CFR 60**. Of particular applicability to Navy installations are:

- Subpart D Fossil Fuel Fired Steam Generators
- Subpart E Solid Waste Incinerators
- Subpart K Storage Vessels for Petroleum Liquids
- Subpart O Sewage Sludge Incinerators

10.18.5 **<u>Resource Conservation Recovery Act (RCRA) Subpart CC (Effective 12/6/95)</u>.** RCRA subpart CC are new regulatory requirements for VOC content in waste and fugitive emissions of VOCs. Depending on container sizes, wastes must be measured for VOC content. This is accomplished by obtaining a sample of the waste and analyzing for VOC concentration using **EPA laboratory method 25D (40 CFR 60)**. Wastes containing greater than 100 ppm total VOC are subject to control equipment and/or management practices to limit emissions of VOCs.

The other aspect of RCRA subpart "CC" pertains to air sampling of large containers and process units for fugitive emissions of VOCs. Large containers, valves, fittings, covers and other areas where leaks can occur must be sampled for fugitive emissions of VOCs in the surrounding air. If total VOC levels exceed 500 ppm, the containers/process units are subject to control devices and/or management practices to eliminate or minimize emissions. VOC measurements are made per **EPA method 21 (40 CFR 60)** using FID, PID, or other approved instrumentation.

10.18.6 **<u>Asbestos</u>**. Several Federal agencies are charged with regulating asbestos products and wastes. The **Occupational Safety and Health Administration (OSHA)** sets limits for worker exposure on the job. The **Consumer Product Safety Commission (CPSC)** regulates asbestos in consumer products. The EPA regulates the management and disposal of asbestos-containing wastes.

Asbestos emissions during building demolition or renovation, as well as the transport and disposal of asbestos waste, are regulated under the EPA's NESHAP. Through NESHAP, the EPA requires prework notices and specific work practices to be used during demolition and renovation operations involving **asbestos containing materials (ACM).**

The Navy itself also regulates asbestos within an installation. The Chief of Naval Operations (**OPNAV**) instruction, OPNAVINST 5100.23, *Navy Occupational Safety and Health (NAVOSH) Program Manual*, Chapter 17, provides guidance for controlling or eliminating exposure to asbestos on shore facilities. OPNAVINST 5100.19, *Navy Occupational Safety and Health (NAVOSH) Program Manual for Forces Afloat*, Chapter B1, provides guidance for forces afloat.

Samplers must be trained and certified for asbestos sampling.

10.18.7 **<u>Emergency Situations</u>.** Air monitoring may be performed during emergency situations such as fires or leaks of hazardous chemicals. Such monitoring is typically done with real time instruments to establish the limits of evacuation zones and to determine the area where respirators must be worn. In addition, air samples may be taken at the perimeter of the evacuation and respirator (exclusion) zones to determine the extent to which people may have been exposed to toxic chemicals.

Although emergency sampling may be required with little or no planning, it must be done very carefully and with complete documentation because of the possibility for injury due to exposure and due to the possible need to use information obtained in litigation.

10.18.8 **<u>Nuisance Complaints</u>.** Nuisance complaints from the general public are primarily related to odors, eye irritation or, in some cases, serious illness. The Sample Analysis Plan must address two major areas of concern: (1) Identity of chemicals involved and (2) Measurement of chemical concentration in the air at the location where the complaints arise. Since odor thresholds for many noxious chemicals are extremely low, and certain individuals are very sensitive to chemicals, the levels of interest may be considerably below regulatory limits or the concentration that will cause acute symptoms. In addition, the complaints may arise only sporadically as the result of certain work at the facility or certain weather conditions. To avoid claims of a cover-up, the planning process must be open to review by those initiating the complaints. All work must be done carefully, with thorough and legally defensible actions and documentation.

WARNING

10.19 <u>SAMPLING EQUIPMENT LIST</u>. Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to air sampling:

- Safety Equipment, as required
- Photo Ionization Detector (PID)
- Flame Ionization Detector (FID)
- Infrared Radiation Absorbance Detector
- Detector tubes (Dräger, Sensidyne, Matheson)
- ChromAir (Gilian)
- Explosimeter
- Indicator Tubes
- Bag Samples (Tedlar®, Teflon®, Mylar® and Saran®)
- Metal Canister Samplers (Summa® polishing process)
- Glass Globe Samplers
- Passive Sampling Badges
- Solid sorbents
- Organic polymers
  - Amberlite® XAD-2
  - Thermosorb/N
  - Polyurethane foam
- Inorganic powders
  - silica gel
  - Porasil®
  - Florisil®
  - alumina
  - inorganic molecular sieves
- Carbon adsorbents
- Chemical Derivation Chemicals
- Cryogenic trap
- Nebulization/Reflux condensation Trap
- Particulate Filters
  - Borosilicate fiberglass filters
  - Cellulose fiber, such as Whatman No. 41 filter paper
  - Glass or quartz fiber
  - Membrane filters (cellulose esters, Teflon®, nylon, etc.)
  - Nucleopore filters
  - Centrifugal Collector

Other Devices Used to Collect Aerosols:
- Wet Collectors
- Electrostatic separators

Equipment for Dry Deposition Sampling:
Denuders
- Cellulose, Glass Fiber, and Polymeric Filters
- Particulates sized on Teflon membrane filters which separate particles under 10 microns (PM10) or 2.5 microns (PM2.5).
- Nylon filters are used to remove NO_2 gas
- Cellulose filters impregnated with 15 weight percent potassium carbonate and 5% glycerol in water for SO_2
- Cellulose filters impregnated with 25 % citric acid and 5% glycerol in water for NH_3
- Cellulose filters impregnated with 25% TEA, 4% ethylene glycol, 25% acetone in 46% water for NO_2

Equipment for Stack Samples
- Method 0010 - Modified Method 5 Sampling Train
- High efficiency glass or quartz fiber filter
- Packed bed of porous polymeric adsorbent resin (XAD-2)

Method 0020 - Source Assessment Sampling System (SASS)
- Three heated cyclones
- Heated high-efficiency fiber filter
- Cartridge containing 150 grams of XAD-2 resin

Method 0030 - Volatile Organic Sampling Train (VOST)
- Glass-lined probe and a Volatile Organic Sampling Train
- Sorbent resin traps
- Impingers
- Resin trap containing approximately 1.6 grams of Tenax®
- Resin trap (back trap) containing approximately 1 gram each of Tenax® and petroleum-based charcoal, 3:1 by volume

NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 11

BIOLOGICAL SAMPLING AND ECOLOGICAL RISK ASSESSMENT

11.1 PURPOSE. This chapter provides guidance for conducting sampling of aquatic life, including assessing organism responses, gathering microbiological data, toxicity testing, and risk assessment.

11.2 SCOPE. The scope includes sampling small streams, rivers, lakes, ponds, wetlands, waste lagoons, and waste effluents.

11.2.1 Assessing Organism Responses. Sampling for floating algal mats; filamentous attached algae (stringy, or with long streamers), bacterial, and fungal growths attached to the bottom or other substrates (rocks, sticks, or some solid surface on which organisms can attach); aquatic vascular plants (plants other than algae); fish; and bottom associated organisms (benthos) are all included in the chapter.

11.2.2 Microbiological Sampling. Sampling for microbiological testing must follow the appropriate regulatory protocol. For the Drinking Water program sampling requirements refer to the Drinking Water Chapter. Other microbiological sampling may include swimming pools, bathing places, industrial discharges for the NPDES program, surface water, ground water, air, filters, and other media. Microbiological sampling and testing of wastewater samples is part of the NPDES program found in 40 CFR Part 136. Microbiological testing of sludge composting is part of 40 CFR Part 503. Sampling of air for fungus, molds, bacteria and viruses are performed when assessing indoor air quality or worker health and safety issues.

Several methods for collecting samples may be used depending on the data needs. All sampling devices, containers or other apparatus coming into contact with the sample must be sterilized or disinfected to ensure that samples are not inadvertently contaminated. Samples are collected using a sterilized or disinfected sampling device and transferred to a container for transport to a laboratory. In cases where microbial populations are difficult to detect, large quantities of the sample are concentrated through filtration and placed on selective media to isolate the organisms. The filter is analyzed by microscopic techniques, selective culture media or using a variety of techniques to isolate the organisms of interest. Samples may be collected on non-selective or selective holding culture media and returned to the laboratory for further isolation and identification. In all cases the volume of source material is quantified to report results based on a consistent sampling volume.

Samples collected to establish data trends should be consistent as to depths, tides, location, name, method of sampling, method of testing, sampling frequency and as appropriate, correlation to turbidity or particulates, rainfall, flow velocities, and seasonal effects.

Samples are collected in the appropriate size and type of sterilized container. Grab samples of water are collected with a minimum sample volume of 100 milliliters. Water samples are placed in containers with at least one inch headspace to allow for sample shaking prior to analysis. Holding times range from six hours to 72 hours depending on regulatory requirements. Samples are stored under refrigeration during transport to reduce growth of the organisms prior to sample analysis. Samples containing disinfectants such as chlorine are collected in containers with a dechlorinating agent such as sodium thiosulfate. Chemical preservation is needed to prevent continued bacterial action and indicate a more accurate count of the microbial content at the time the sample was collected.

11.2.3 Toxicity Testing. The majority of toxicity sampling is performed as part of wastewater compliance with the National Pollution Discharge Elimination System (NPDES) program. Toxicity tests for Chronic Toxicity or Acute Toxicity are specified for the type of water being measured, such as marine, estuarine or freshwater. Toxicity samples, sometimes referred to as bioassay samples, are collected from any one of the following locations, the effluent specified in the NPDES permit, a location between the final treatment and the discharge point, a location prior to final chlorination, the influent to a treatment plant, separate wastewater process streams or non-contact cooling water.

The objectives of the test must be clearly defined and an understanding of the short and long-term operations and schedules must be considered when determining the type of sample to collected. Grab or composite samples are taken based on the type of wastewater characterization required, whether the discharge is intermittent or continuous, and the logistical problems associated with sample collection.

Sampling handling should be minimized to ensure that significant changes do not affect the representativeness of the sample. Aeration during collection and transfer should be minimized to reduce the loss of volatile chemicals. Sample containers should be completely filled leaving no air space between the contents and the lid. Sample volumes range from one liter to four liters depending on the test selected.

Samples should be chilled as they are collected and held at 4^{o} C until used. Chilling the samples inhibits microbial degradation, chemical transformations and loss of highly volatile toxic substances. Chlorine residual is measured immediately following sample collection. Sample holding time for acute and chronic toxicity is 36 hours for off-site tests and 24 hours for on-site tests. Variances may be granted by the state authority as long as supportive data shows volatilization or sorption of toxins on the sample container surfaces does not reduce the sample toxicity.

The number and frequency of renewal samples are specified in permits or study protocols. Sufficient sample must be collected to perform the toxicity test and the related chemical tests. Chemical tests are performed to determine the general water quality characteristics of the samples. These may be compared later to known stream conditions to evaluate deterioration or possible sources of toxicity. The chemical tests should be sampled and preserved following standard NPDES program criteria.

Sample collection must be coordinated with the laboratory. Tests must commence within the specified holding times. The EPA recommends the test start on the day of arrival at the laboratory. Test organisms for controls must be prepared by the laboratory starting at least one week prior to receiving the sample. Laboratory notification should be at least two weeks prior to sample delivery. The laboratory should be called when the samples are shipped and told of the estimated time of arrival. When the samples arrive at the laboratory they are logged in and the temperature is measured and recorded. Samples are to remain at 4^{o} C until the samples are used. Sampling for bacteria, viruses, and protozoa requires the collection of water samples while employing precautions against sample contamination. The laboratory can provide information on the type and quantity of samples needed.

11.2.4 Risk Assessment. Knowledge of the relative number and kinds of organisms that live in a reach of water provides a basis for evaluating pollution risk to the ecosystem, to fish and shellfish, and to man, who may use the water for drinking, a source of food, or for recreation.

11.3 HAZARDS AND SAFETY PRECAUTIONS.

Where applicable, see **Chapter 3, Section 3.3.3**.

11.4 SAMPLING CONDITIONS.

11.4.1 Flowing Water. Biological considerations in streams and rivers include sampling for fish, bottom and other substrate associated organisms, and filamentous attached growths. Suspended algae sometimes are used but they flow with the current and are difficult to assess, unless one is an expert in identification of algae.

Artificial substrates sometimes are used in stream reaches where physical conditions may not present an adequate habitat for colonization of bottom associated organisms. The artificial substrates provide such a habitat at the site where they are anchored.

Although fish may be transient, they are an important consideration in stream sampling. Fish represent one of the end products of the aquatic phase of the food chain. However, because of their mobility, they indicate water quality only at the particular time of capture. Fish samples may be collected by nets, poisons, and electrofishing.

11.4.2 Standing Water. Biological considerations in lakes, ponds, wetlands, and waste lagoons include sampling for suspended algae and bottom associated organisms. Fish should be sampled if practical, but it often is not practical to obtain the equipment necessary for fish sampling. This equipment for lakes generally consists of large nets, experimental gill nets, or hoop nets. Floating algal scum and filamentous algae in shallow water along shorelines should be qualitatively sampled for the purpose of identification. Submerged and emergent, rooted and floating, vegetation should be identified and the general location and extent of growth noted on an appropriate site map.

11.4.3 Qualitative Sampling. Qualitative sampling determines the variety of species (kinds of organisms) occupying a reach of waterway. The qualitative search for bottom associated or substrate associated organisms in rivers and streams includes rocks, gravel, sediment or sludge, plants, submerged twigs or debris, or leaves of overhanging trees that become submerged and waterlogged. It is convenient to scrape and wash organisms from these materials into a bucket partially filled with water and then pass this water through the U.S. Standard No. 30 sieve to concentrate and retain the organisms. The collected sample may be preserved for organism sorting and identification later. The investigator should search until there is certainty that the majority of species in the sampling area have been collected. In some environments, it is possible only to collect qualitative samples because the physical nature of the waterway may be such that quantitative sampling is not feasible.

The entry in the log book should note that 30 minutes were spent in collecting a qualitative sample of organisms from isolated rocks on the stream bed, and from waterlogged twigs and submerged leaves from overhanging tree branches along the stream banks.

Filamentous growths of green algae, fungi, and bacteria are often found in rivers and streams enriched with nitrogen, phosphorus, and sugars. Finding such growths provides another indicator of water quality, which the biologist can use in assessing waterway conditions. Generally, it is sufficient to qualitatively collect such growths and place them in a sample jar with preservative for later identification.

Suspended algae can be quantified, but it is a laborious task. Generally it is sufficient to identify the major components of an algal population and indicate the relative abundance of those components. This requires only that a l-liter water sample, containing the algae, be collected and preserved for later identification.

11.4.4 Quantitative Sampling. Quantitative sampling involves a sample that can be associated with a volume of water for suspended algae, or with the sediment surface area for bottom associated organisms. A quantitative bottom sample for organism identification and determining relative abundance can be obtained by any of the deep water dredges, such as the Ponar dredge or Ekman dredge, and the Surber stream sampler. Quantitative determinations also can be obtained from samples for microbiological testing, as well as bioassays for toxicity determination.

11.5 PREPARATION.

11.5.1 Assessing Organism Responses. For field species identification and ecological assessment, samplers should be accompanied by a scientist experienced in field biology to assist in performing sampling and assessment of site conditions.

There are, however, general principles to be applied when preparing to collect biological samples. Upon approaching a sampling location, a number of observations should be noted and recorded in the field log book. These observations may be required later in assessing biological findings. Observations are made on water depth; presence of riffles and pools; stream width; flow characteristics; bank cover; presence of slime growths, attached algae, scum algae, and other aquatic plants; any organisms noted; and unusual physical characteristics such as silt deposits, sludge deposits, iron (brown staining) precipitates, or noticeable waste material.

An example entry in the log book follows: May 15, 1996, 0900 hours, Sampling location - Rapid Run Creek; 500 feet due west of State Highway 252 crossing; stream 50 feet wide; water 18 inches deep; swift current; substrate composed of sand, gravel, and fist-sized rough rock; one Surber Stream Bottom Sample collected from mid-stream; qualitative sample from submerged brush near shore also collected; no sludge or silt deposits; no algae noted; minnows noted but not identified.

11.5.2 Microbiological Sampling. Preparation for collecting samples for bacteria, viruses, and protozoa requires precautions to ensure that sterile collection bottles are used and that sampling procedures will not contaminate the collected samples. The examining laboratory should provide the collection bottles, as well as guidance on sample collection. Collected samples may need to be cooled, and should be returned to the laboratory as rapidly as possible for examination.

11.5.3 Toxicity Testing. Testing effluents for toxicity to test organisms may involve bringing test equipment to the site, however, certain bioassay tests can be performed when samples are properly collected and brought to the laboratory. This is another area in biological testing where experienced or certified laboratories with proper equipment and healthy test organisms should be consulted or employed to perform the required tests. Many States certify biological and chemical laboratories for permit testing purposes.

11.5.4 Risk Assessment. Preparation for risk assessment requires an understanding of the water ecosystem, including chemistry, toxicology, organism interrelationships, food webs, sediment transport, persistency, bioaccumulation, and organism purging.

11.6 SAMPLING PROCEDURES.

11.6.1 Assessing Organism Responses. The species composition and relative population of fish and bottom associated organisms provide information on the relative "health" of the environment in which they live. Stream organisms can be divided into those that are sensitive and require clean water, such as stonefly nymphs; those that are moderately tolerant of pollution, such as many leeches or sowbugs; and those that are very tolerant of pollution, such as sludgeworms. Organisms that live on riffles in streams (shallow rocky areas with strong current), and in bottom sediments, do not travel great distances during their life spans of two or more years. Thus, the presence of these organisms provides a history of water quality for a lengthy period.

This is as demonstrative test; it demonstrates the effects of pollution on a receiving waterway.

11.6.1.1 **Plankton Net.** Samples of water for suspended algae and other plankton are obtained in much the same manner as samples of water for chemical analyses. In most cases, a 1-liter volume of ambient water is sufficient. Unless the samples are examined soon after collection they must be preserved with either 4 percent formalin or one of the special plankton preservatives.

A plankton net (**Figure 11-1**) is useful in concentrating algae and other plankton when the population is sparse. It can be towed behind a boat for a specified distance. A recorded volume of water can also be poured through it. In either case, the fine-meshed net will retain microscopic organisms, which are washed down the interior of the net into a collection vial at the end.

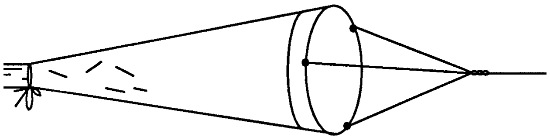


Figure 11-1 Plankton Net

Advantages:

- Concentrates microscopic organisms from water

Disadvantages:

- Towing behind a boat may sample only a portion of the volume of water because the fine mesh of the net creates flow resistance
- More time consuming than collecting a sample of water for later laboratory concentration

11.6.1.2 **Kemmerer Depth Sampler.** Used to collect algal and other plankton sample at any water depth. For description, see **Section 7.5.1.5** and **Figure 7-4**.

11.6.1.3 **Ekman Dredge.** Used to collect bottom associated organism sample in non-flowing or sluggish waters with soft sediment or sludge bottoms. For description and use, see **Section 10.5.1.2** and **Figure 10-1**. After the dredge is closed, it is brought to the water surface and the contents are placed in a large pail or tub. Water for sample dilution is added, and the sample is mixed into a slurry with the slurry being passed through a U.S. Standard No. 30 sieve while the sieve is being rotated in the water. The washing operation is repeated until all fine material has passed through the sieve, and all organisms are retained in the sieve. The organisms and coarser debris are then removed from the sieve and are preserved.

Wide-mouthed, tapered pint freeze jars have proven to be excellent bottom organism sample containers. When these jars are filled half full with 10-percent formalin before the day's activities of sample collection, it is a time-saving process to transfer the concentrated sample from the side of the sieve to the jar of preservative by lightly tapping the sieve against the top of the jar. The investigator is assured always of a minimum of 5 percent formalin in the sample container, which is a sufficient strength to preserve the collected organisms. After the samples are preserved in the field they are brought to the laboratory where the organisms are separated from the debris, placed in respective groups, identified, and counted.

11.6.1.4 **Ponar Dredge.** Used to collect bottom associated organism sample. For description, see **Section 10.5.1.3** and **Figure 10-2**. Collect the sample in a manner similar to the procedure for sample collection with the Ekman dredge.

11.6.1.5 **Surber Stream Bottom Sampler.** To sample riffle areas in streams, a Surber square-foot stream bottom sampler (**Figure 11-2**) is widely used. It consists of two 1-foot-square brass frames hinged together at right angles; one frame supports the net which is held extended downstream by current velocity, the other encloses the sampling area. In operation, the sampler is so placed that organisms dislodged by hand from the substratum within the sampling frame will be carried into the net by the current. In stagnant or in slowly moving water, it often is not practical to employ this sampler.

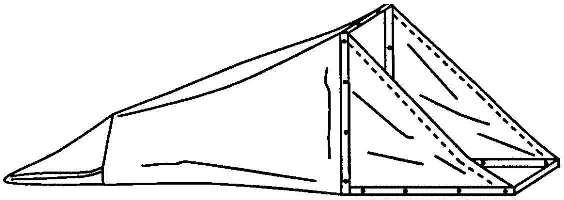


Figure 11-2 Surber Stream Bottom Sampler

Advantages:

- Rapid, effective means of collecting bottom organism sample
- Sample is concentrated and ready to be put into collection jar

Disadvantages:

- Can only be used in wadable streams where the sampler's hand can easily reach to the stream bottom

- When substrate is composed of larger than fist-sized rocks, sampler will not collect quantitative sample
- Sampler will not function properly in water with sluggish current

11.6.1.6 **Electrofishing Equipment**. Electrofishing is conducted by means of an alternating or pulsed voltage applied to water using a positive and negative electrode. The fish are attracted to the positive electrode. An electrical field is of sufficient potential to demobilize the fish near the positive electrode, but decreases in intensity with distance. Within a short time, demobilized fish will recover, unharmed.

CAUTION

Use due caution when working with electricity and water

The electrofishing unit may consist of a 110 volt, 60 cycle, heavy duty generator; an electrical control section, which is a modified commercially-sold variable voltage pulsator; and electrodes. The electrical control section provides selection of voltages from 50 to 700 volts AC and 25 to 350 volts DC. The variable voltage allows control of field size in various types of water.

Electrofishing can be performed by personnel protected by waders in shallow waters, or from a boat.

WARNING

When a boat is used, the front portion should have a constructed deck on which two fish collection personnel may stand. The deck should have a sturdy waist-high rail that will protect personnel from accidentally falling into the water should the boat operator suddenly reverse the motor.

An appropriate "dead-man" switch is installed on each electrode handle when used in wading, as well as individual foot switches for the fish collectors when the electrodes are attached to the boat. These switches must be depressed for the current to flow through the electrodes.

When electrofishing in estuarine (slightly saline) waters, it is often useful to perform the sampling at night with the use of lights on the front of the boat. Fish collection under these conditions is more effective.

Advantages:

- Permits collection of fish for identification, weighing, measuring, and scale sample collection without injury to the fish
- Effective in waters less than about 20 feet deep

Disadvantages:

- Requires a minimum of 2 persons, and preferably a 3-person crew to adequately operate
- Generally not suitable for pond or lake environments, except in near-shore areas

11.6.1.7 **Multiple-plate Artificial Substrates.** Artificial substrates have been successfully employed for collecting bottom fauna in flowing streams, especially where the bottom substrate is composed of sand, bedrock, or other material that is not conducive to other sampling procedures. One multiple-plate sampler constructed of tempered hardboard (Hester-Dendy type, **Figure 11-3**) has been especially useful. This sampler is constructed of eight 3-inch squares, separated by seven l-inch squares, and held in place by a bolt or threaded rod. When constructed in this manner, the sampler exposes slightly more than one square foot of surface to which organisms can attach.

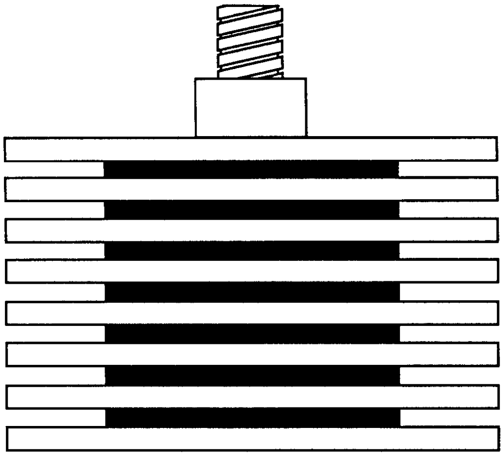


Figure 11-3 Multiple-plate Substrate

Artificial substrates are placed in the water for 3 to 6 weeks and then carefully removed to prevent losing the organisms that have made them a temporary home. The substrates should be placed at similar depths and in similar physical relationship to the stream at all locations. Usually they are placed about 1-foot beneath the water surface or 1-foot off the stream bed.

NOTE:

Loss of some substrates because of vandalism or flooding should be anticipated.

Advantages:

- Provides a convenient means of collecting organisms from areas with otherwise poor habitat for organisms
- Provides quantitatively comparative sample because substrates offer the same organism colonization area

Disadvantages:

- Requires two trips to sampling area: one to set the samplers in the water, and one to collect the substrates
- May result in loss of some data and equipment (Equipment is left unattended for several weeks.)

11.6.2 Microbiological Sampling. Sampling for bacteria requires great care to ensure that the sample does not become contaminated. The sample collection bottle must be sterile. Care must be taken to ensure that the fingers of the sample collector do not touch the inside or lip of the sampling bottle or inside of the lid. In flowing water, the opened sample bottle should face upstream. Generally, a sample collected from the mid-stream area of flowing water is adequate.

When sampling streams by wading, or when sampling from a boat in lakes and ponds, the sample collector faces upstream, and the sample is collected by opening the sample bottle and collecting the sample upstream from the sample collector by a scooping motion that thrusts the sample bottle into the water and up when the bottle fills. The lid is replaced on the sample bottle with care to ensure that sterile conditions are maintained. When sampling from a bridge, a weighted sample bottle holding device is used to lower the sample bottle into the water. Care should be taken to ensure that the sterile sampling bottle is upstream from the holding device to preclude contamination from the holding device from entering the sterile bottle.

Coliform bacteria are often used as indicators of pollution. They are found in the intestinal tract of all warm-blooded animals, but they are found also in other situations. A more positive identification of a health hazard is the further identification of fecal coliform bacteria. If the purpose of sampling is to comply with drinking water requirements or specific permit conditions, the sampling protocols will be detailed in the approved method.

In cases where microbial populations are difficult to detect, large quantities of a sample are concentrated through filtration and placed on selective media to isolate the organisms. The filter is analyzed by microscopic techniques, selective culture media, or using a variety of techniques to isolate organism of interest. In all cases the volume of source material is quantified to report results based on a consistent sampling volume.

Sampling for algae in lakes, ponds, or reservoirs may be for purposes of identifying the predominant species, or for obtaining a quantitative count. Samples may be collected from different water strata vertically, as well as several identifiable locations in the lake or pond.

11.6.3 Toxicity Testing. Toxicity testing involves exposing an identified test organism to a known concentration of a potential toxicant for a defined time under controlled conditions and observing the organism response. Generally, discharge permits require the testing of whole effluent in a series of dilutions to determine the concentration in which there is a mortality of 50 percent of the test organisms. Usually, also, the test is required on more than one test organism, and a species of fish and an invertebrate species often are used. Most permits specify the test organisms to be used. Also, the regulatory authority has specific procedures for conducting the toxicity test.

11.6.3.1 Acute Toxicity Testing. An acute toxicity test is a short term test of 24, 48, or 96 hours. The test may be performed as a static test in which test organisms are placed in a known concentration of an effluent or a potentially toxic substance. Temperature, dissolved oxygen, pH, and number of surviving organisms are recorded at the beginning of the test and each 24 hours thereafter.

A static renewal test may be used in which the test organisms are transferred every 24 hours to a fresh solution containing the same mixture concentration as the initial 24 hours. Dead test organisms are removed as soon as noted. Temperature, dissolved oxygen, pH, and number of surviving organisms are recorded at the beginning of the test, each time before test solutions are changed, and at the end of the test.

11.6.3.2 Chronic Toxicity Testing. Chronic toxicity tests are longer term tests, generally lasting seven days or longer depending upon the test circumstances. They may involve flow-through tests in which equipment provides for a continuous flow of a predetermined mixture of effluent or potentially toxic material and dilution water through the test chamber holding test organisms. Generally, the dilution water is taken from the receiving waterway.

11.6.3.3 Caged Organism Testing. Placing appropriate test organisms in suitable cages upstream and downstream from an effluent or a suspected source of pollution is a convenient way of determining toxicity within the receiving water. Fish known to inhabit the waterway may be used as test organisms, and at the end of the test, fish tissues may be examined to determine bioaccumulation of the toxic substance. Large invertebrates, such as stonefly or mayfly nymphs or caddisfly larvae may be used as test organisms, and small pieces of pipe with wire mesh covering the ends may be used as suitable cages. Potential cannibalism within the test organism community must be avoided. These procedures require collecting a sufficient number of organisms to be used as test organisms.

11.6.3.4 Sediment Testing. Sediments can be used as a substance to be tested. Specific procedures for testing dredged spoil toxicity have been developed by the U.S. Corps of Engineers and Environmental Protection Agency. Generally, three inches of sediment to be tested is placed in a test jar, unpolluted water is carefully introduced above the sediment, the suspended solids are allowed to settle, and a known number of test organisms are introduced. Test organisms may be those from an unpolluted portion of the receiving waterway, such as burrowing mayfly nymphs, small clams, or midge larvae. Temperature, dissolved oxygen, and pH of the superimposed water should be recorded at intervals during the test. This test may be continued for several days, and some tests have been continued for 30 to 60 days. At the end of the test, the live test organisms are recovered through sieving the sediment.

11.6.3.5 Bioaccumulation Determination. The bioaccumulation potential of specific toxic substances is an important consideration in the investigation of any waterway. These potentials often are recorded in the literature for many specific elements and compounds. They may be determined in any investigation by sacrificing surviving test organisms that have been exposed to a toxic substance, and chemically testing the organism flesh. The bioaccumulation within the whole organism, in which the ground up organism is tested, is often separated from the bioaccumulation in the fatty tissues alone. It should be noted that bioaccumulation represented the sum of a specific element or compound absorbed by an organism from the water, as well as the food ingested, which may have increased the bioaccumulating substance concentration as it has passed up the food web.

Surviving caged organisms may serve as organisms on which to determine bioaccumulation, providing they had not bioaccumulated the specific substance prior to being selected for the caged experiment. Otherwise, organisms residing in the reach of waterway of interest may be captured for the bioaccumulation determination.

11.7 QUALITY ASSURANCE, QUALITY CONTROL.

Quality assurance in the collection of field biological samples is based primarily on the demonstrated integrity of the sample collector. Biological sampling equipment must be used in its intended manner. Every effort must be made to ensure that the collected sample is representative of the ecosystem from which it was collected.

All samples must be accompanied by a complete **Chain-of-Custody Record**. Main elements of the Quality Assurance Plan (QAP) for biological sampling are:

- A clear objective
- Sampling procedures
- Preservation techniques
- Sample custody
- Analytical procedures
- Quality Control parameters
- Equipment check
- Performance audit
- Quality assurance report
- Corrective action

11.8 SAMPLING EQUIPMENT LIST.

Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to biological sampling:

- ☐ Sterilized sample containers with or without dechlorinating agent
- ☐ Disinfection solution(s)
- ☐ Wide-mouth jars for bottom organism samples
- ☐ Preservation chemicals and reagents
- ☐ Formalin solution or alcohol for sample preservation
- ☐ Kemmerer Depth Sampler, with rope
- ☐ Ponar Dredge, with rope
- ☐ Ekman Dredge, with rope
- ☐ Plankton Net
- ☐ Surber Stream Bottom Sampler
- ☐ Multiple-plate artificial substrates
- ☐ Electrofishing Equipment
- ☐ Flat-bottomed boat, with motor
- ☐ Waders
- ☐ Large bucket or small tub to contain dredge samples
- ☐ U.S. Standard No. 30 sieve
- ☐ Forceps

11.9 Ecological Risk Assessment. The U.S. EPA defines Ecological Risk Assessment (ERA) as "the process that evaluates the likelihood that adverse ecological effects may occur or are occurring as a result of exposure to one or more stressors" (USEPA, 1992). ERA is a relatively new, multi-disciplinary, and rapidly evolving set of procedures. If an ERA is required or indicated at your installation, you should recognize that it will entail a planned technical approach, with much attention to the problem definition, in which the sampling methodology is a relatively small but necessary piece.

The ERA process begins by identifying the stressors (e.g., the release of a toxic chemical into surface waters) and then using a series of measurement and test procedures, often with statistical modeling, to evaluate the relationship between the stressors and the potential effects (Suter, 1993).

The purpose of this section is to introduce ERA principals and provide preliminary guidance on when and how to use the ERA process. More detailed guidance may be found in the references section.

11.9.1 Legislative and Regulatory Drivers. The initial legislative impetus for ERAs came from the National Environmental Policy Act (**NEPA**, 1969). This Act required federal agencies to consider the environmental impact of their proposed actions. Since then, a number of environmental laws have used the principals of ERA to establish regulations, criteria, and standards, including the Clean Water Act (**CWA**), Clean Air Act (**CAA**), and the Marine Protection, Research, and Sanctuaries Act. Most recently the EPA has promulgated protocols to address the environmental risks of past hazardous waste sites under the Comprehensive Environmental Response Compensation and Liability Act (**CERCLA**, or **Superfund**). In particular, the Remedial Investigation/Feasibility Study (**RI/FS**) process of CERCLA requires the characterization of the nature and extent of contamination and the resulting human and environmental risks posed by the site (USEPA, 1988.)

Human health risk assessment, which dates to the early 1980s (National Research Council, 1983) established the structured process that became the initial framework for an ecological risk assessment. The EPA published its first guidance on ERA, *Review of Ecological Risk Assessment Methods*, (USEPA, 1988). Thereafter, the EPA stepped back to reassess the overall process. It convened a series of workshops with highly experienced environmental scientists to develop a framework specifically for ERA (USEPA, 1992). During these early stages, responsible parties under CERCLA, or under the Navy's Installation Restoration (**IR**) program, began learning ERA by doing it and incorporating EPA guidance as it was published. The application of ERAs increased with the advent of Corrective Actions under the Resource Recovery and Conservation Act (**RCRA**) that were designed to address hazardous waste sites created after the cut-off date (about 1980) for action under CERCLA. Since 1992 the EPA has also incorporated ERA into the implementation of the Toxic Substances and Control Act (**TSCA**) and the Federal Insecticide, Fungicide, and Rodenticide Act (**FIFRA**) to provide protection from "unreasonable [environmental] risks" (Suter, 1993). The EPA is now finalizing the first detailed set of guidelines for conducting ERA. The draft of this document has been out for public review since October 1995 (US EPA, in prep).

11.9.2 Benefits of ERAs. The potential benefits from conducting ERAs include:

- Correctly identifying harm, ecological risk can reduce the cost of unnecessary or overprotective remediation that may cause further environmental damage (e.g., habitat destruction)
- Providing data to support environmental programs besides CERCLA, such as RCRA, CWA, dredging, MPRSA, natural resource conservation, and NEPA
- Developing a baseline ecological database that can be used for a long-range environmental profile of the facility
- Providing data for the decision-making associated with managing ecological risks, including:
    - Comparing, ranking, and prioritizing risks
    - Analyzing uncertainty to lend credibility to the assessment and focus on information gaps
    - Predicting the outcomes from remediation alternatives, thereby assisting decision-makers who must evaluate tradeoffs and alternative risk scenario

11.9.3 ERA Protocol. Assessing the risks to an ecosystem as a whole requires the development and implementation of complex methodologies that consider spatial and temporal variability. An ERA requires:

- An understanding of the movement and transformation of pollutants and other stressors within the environment and into the air, water, soil, sediment and biota
- An assessment of potential effects caused by those stressors
- A Characterizationing of the risk and an evaluation of the weight of evidence of an effect

The outcome of the risk assessment can be used in setting policy and analyzing economic costs and benefits.

Although formal national guidance for conducting ERAs has not yet been finalized by the EPA, regional EPA offices and states are implementing these studies based on the EPA's *Framework for Ecological Risk Assessment* (USEPA, 1992), which includes three phases: Problem Formulation, Analysis, and Risk Characterization (see **Figure 11-4**).

Problem Formulation. This first phase defines the problem, determines the assessment endpoints (the ecological values to be protected at the site) and measurement endpoints (a measurable ecological characteristic that is related to the assessment endpoint), and lays out a plan.

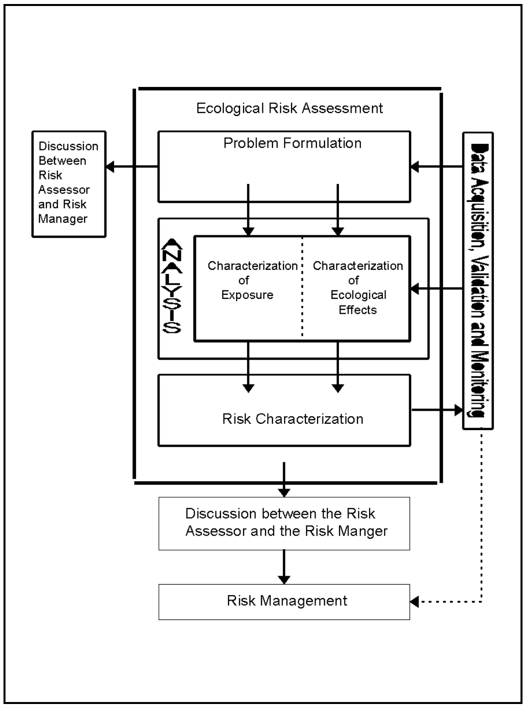


Figure 11-4 Framework for Ecological Risk Assessment

To define the problem, all available information about the site is gathered, including source and stressor characteristics, ecosystems potentially at risk, and any observed ecological effects. To select the proper assessment endpoints, three criteria should be met:

- the endpoint has ecological relevance within the affected ecosystem
- the receptor exhibiting the endpoint is susceptible to the stressor(s)
- policy and societal goals have been considered

The final products of Problem Formulation are a Conceptual Model, consisting of testable hypotheses to explain how the stressors may affect the ecosystem, and an Analysis Plan.

Source: USEPA, 1992

Analysis. This phase involves characterization of exposure and of effects. The former involves determining of the spatial and temporal distribution of pollutants of concern, and their interaction with the ecosystem. It evaluates the interaction of the stressor with one or more ecological components. Stressor characterization considers not only the primary stressor but also secondary stressors that can arise as a result of various processes.

Characterization of ecological effects involves determining the impact of pollutants on individuals, populations, and communities. It evaluates adverse effects of a stressor on a population or community under a particular set of circumstances.

Risk Characterization. Risk characterization takes the information developed in the analysis phase to make a statement about the likelihood that exposure to chemical stressors is producing or may produce adverse ecological effects. Because methods for quantifying risk are not widely available, ERAs usually develop conclusions based on a "weight-of-evidence" whether there are ecological effects or not. Although this may not result in a hard-and-fast number, the result can be used in the risk management process to prioritize and rank remedial alternatives and optimize the environmental benefit of costs expended.

11.9.4 New EPA Guidelines for ERA.

The EPA's "Draft Proposed Guidelines for Ecological Risk Assessment" builds on and expands the original three steps. Improvements being considered by the EPA are mainly in the problem formulation and risk characterization areas. Problem formulation expands the interactions between the risk assessor and risk manager that help define the purpose, boundaries, and resource limitations of the assessment, delimiting ecological values, goals, and assessment endpoints, preparing the conceptual model, and developing an analysis plan. Within risk characterization, key elements include estimating risk and evaluating ecological significance, using a weight of evidence, and calculating uncertainty levels to the degree possible. It is anticipated that the new EPA guidance document for ERA will be published in 1997.

ERA requires careful planning before initiating the work effort. Problem formulation, which should include development of testable hypotheses, is key to a successful ERA. To the degree possible, ERAs should be designed to provide high quality quantitative data that evaluate definable risks. It is important that the risk assessment and assessors are separate from the risk management process because the latter must weigh economic, social and operational considerations as well as ecological risk.

11.9.5 Tiered Approach to ERAs. Without detailed and careful planning ERA's can become costly and complex studies that may not yield appropriate information to evaluate risk. Since the potential toxicity or impact of sites or stressors is likely not known prior to conducting an assessment, a tiered or phased approach to conducting ERAs has been advocated by the EPA and others. If initial studies indicate that there is a likelihood of ecological risk, then progressively more complicated and complex assessments are done.

Typically the first step in a tiered approach is a "screening level" assessment that involves limited sampling of contaminated media, calculation of hazard quotients and an evaluation of the site's habitat. These screening procedures can help to identify the specific ecological habitats that could be impacted by the contaminants at a site. The result should be fewer receptors that would need to be evaluated for the site. It should also provide the basis for further evaluation (i.e. Problem Formulation).

It is useful to consider that for any given site (or facility) a large number of possible receptor (ecological component exposed to a stressor) and contaminant pathways exist.

The initial step in the screening procedure should be to reduce the possible pathways and identify the important receptors and pathways that will be necessary to define the ecological risks. There are several approaches to accomplishing this including, literature review, exposure models or direct measurement receptor exposure to the stessor. One cost effective option could be the use of off-the-shelf receptor and exposure pathway models that are selected based on the characteristics of the habitats at the site.

For example, if the site were near a freshwater stream or pond, then an aquatic exposure model should be considered. If the site were associated with coastal or estuarine resources, than an estuarine exposure model should be considered. Obviously, sites without the potential to impact aquatic resources would not need to evaluate those exposure pathways.

This approach may seem overly simplified, but the fact is that much redundant work and lost time can be avoided if off-the-self exposure models were available for screening purposes. The models would need to be fairly generic (i.e., could be applied on a national basis), however, specific endpoints and benchmarks could be developed for use on a regional basis.

As an illustration, consider the estuarine and coastal wetlands exposure pathway developed for Portsmouth Naval Shipyard, and the coastal uplands exposure pathway developed for Naval Air Station Whidbey Island. Similar exposure pathways exist for all facilities that have similar habitats. The generic models focus on specific trophic levels (feeding guilds) and types of ecological receptors present in those habitats. The specific applications of the models would require identifying specific receptors and benchmarks applicable to the site being screened.

Where possible, the models would contain food chain transfer functions to relate exposure levels to tissue concentrations. In other cases, the models would define the exposure point concentrations that need to be developed for the risk assessment. Some possible generic models include: Inland Meadow, Inland Desert, Inland Forest, Inland Wetlands (fresh water lakes, streams, and wetlands),Coastal Plain (forest, grassland, marsh), Coastal Wetland (saltmarsh, intertidal, eelgrass) Estuarine/Marine (bays, harbors, inlets, etc.).

Table 11-1 provides examples of estuarine and coastal wetlands exposure pathways and screening criteria for three case studies from different regions of the country.

The receptors need to be coupled with ecological benchmarks developed for each specific region. In the process, data gaps would be defined that will suggest hypotheses to be tested by the ERA. Site investigations should be identified to determine if receptors are being harmed (e.g. determining the health and status of eelgrass beds adjacent to the site). The procedure would identify field and laboratory studies being conducted that will help develop benchmarks for use in future ERAs. By using this process, chemicals may not be screened out, but unnecessary work will be eliminated. This will reduce the time required to complete the ERA for the RI/FS or other regulatory requirement.

11.9.6 Quality Assurance/Quality Control (QA/QC) Criteria. The following are the minimum QA/QC criteria that should be included in an analytical chemistry contract, especially if the contractor will be analyzing tissues and sediments. Having this information delivered to you is the only way to ensure the Navy obtains scientifically sound and legally defensible data. This approach has been validated by field use in several case studies and has been acceptable to regulatory agencies.

Table 11-1 Example of Screening Criteria and Possible Receptors Exposed in Pathway

Endpoint	Northeast (NE)	California (CA)	Puget Sound (PS)
Sediment	EYRE-L; EYRE-M; Sed. Quality Criteria; Regional Background from EMAP-VP	EYRE-L; EYRE-M; RWQCB (e.g., SF Bay Ambient Values)	WA Sediment Management Standards
Water	Ambient Water Quality Criteria	CA Ambient Water Quality Criteria	WA Water Quality Criteria
Macro-benthos	Amphipod (Ampelsica abdita)	Amphipod (Rhepoxynius abronius)	Apparent Effects Threshold
Plankton	Phytoplankton	Phytoplankton	Phytoplankton
Bottom Feeder	Flounder; Lobster	Sole; Crab	Ling Cod; Crab
Filter Feeder	Blue Mussel	CA Mussel	Blue Mussel
Carnivore	Cormorant; Sea Gull	Brown Pelican	Blue Heron
Top Carnivore	Osprey	Peregrine Falcon	Eagle
Aquatic Plants	Eelgrass; Salt Marsh; Fucoid Algae	Eelgrass	Kelp
Water Fowl	Black Duck; Canadian Goose	Mallard; Black-necked Stilt	Mallard

Each batch of sample material for each matrix (e.g. tissue and sediment) at a minimum should contain:

- Reagent Blank
- Laboratory Duplicate
- Laboratory Fortified Sample Matrix
- Standard Reference Material (SRM)

Table 11-2 presents an example of how to set up a "batch". The QA/QC samples are treated just as field samples and carried through the complete analysis process (e.g., acid digestion for metal analysis, solvent extraction for organics).

The Marine Environmental Support Office (MESO) has a publication which provides more detailed information. This QA/QC procedure was optimized for marine/estuarine systems (MESO, 1992).

11.9.7 Data Management. Data management is an essential adjunct to environmental measurements, in particular measurements required for an ERA. Natural environmental processes can have very broad spatial extents and turnover rates on the order of decades. Most risk-based assessments must supplement the measurements they make with measurements made by other investigators, often for quite dissimilar purposes. Such comparisons may not be possible if the other investigators did not fully document their primary measurements and record the data in digital form. You must do the same with the measurement data you collect. Moreover, you should incorporate the plans for managing the data into the planning what to measure, how to make those measurements, etc. Key (1996) has discussed these issues at length. MESO has prepared a draft data reporting specification based on model discussed by Key. This specification includes a detailed discussion of what attributes to record and how to organize primary environmental measurement data. The Federal Government has adopted standards for recording metadata (i.e., data about data) for data sets of environmental measurements. While the MESO document cited above incorporates many of these same elements into a common data model rather than storing them in separate data files, you should be aware of the Federal standard found in the *FR* 48 (191):30503.

11.9.8 References. The following references provide in-depth information about ecological risk assessment:

Key, G.S.; 1996, "Some Experiences Developing a Generalized Environmental Data Model", Volume 1, pp 101-111, *Proceedings Oceanology International '96 Conference, Brighton, UK*, March 5-8 1996.

Marine Environmental Support Office (MESO), 1992, "Analytical Chemistry Quality Assurance And Quality Control Protocols, Criteria, And Corrective Action For The Estuarine Ecological Risk Assessment At Naval Shipyard Portsmouth, Kittery, Me." NCCOSC RDT&E Division Technical Document 2296.

Table 11-2 Minimum Number of QA/QC Samples and Sequence of Analysis for 16 Field Samples

Minimum Number of Samples			Sequence of Analysis		
Sample	Number	Description	Sample	Number	Description
S1, S2,...,S16	16	Field Samples	CC1	1	Calibration Check
SRM	1	Standard Reference Material	ES1,...,ES10	10	Extracted Samples
LD	1	Laboratory Duplicate	CC2	1	Calibration Check
LFSM	1	Laboratory Fortified Sample Matrix (Spiked Sample)	ES11,...,ES20	10	Extracted Samples
Blank	1	Reagent Blanks	CC3	1	Calibration Check
Total (ES1, ES2,...,ES20)	20	Extracted/Digested Samples	Total	23	Analytical Analyses

National Research Council, 1983, *Risk Assessment in the Federal Government: Managing the Process*, Committee on the Institutional Means for Assessment of Risks to Public Health, National Academy Press.

Suter II, G., et al, 1993, *Ecological Risk Assessment*, Lewis Publishers, Chelsea, Michigan.

U.S. Environmental Protection Agency, in prep, "Draft Proposed Guidelines for Ecological Risk Assessment", Draft EPA/630/R-95/002, (Draft Dated October, 1995).

U.S. Environmental Protection Agency, 1992, *Framework for Ecological Risk Assessment*, EPA/630R92001, Risk Assessment Forum, Washington D.C.

U.S. Environmental Protection Agency, 1988, "Review of Ecological Risk Assessment Methods", EPA /230-10-88-041.

Department of the Navy, Naval Engineering Command, Northern Division, "Draft Final Estuarine Risk Assessment Portsmouth Naval Shipyard Kittery, Maine", 1995.

Mueller, C.; Munns, Jr. W.R.; et al; 1992, "Standard Operating Procedures and Field Methods Used for Conducting Ecological Risk Assessment Case Studies", Naval Construction Battalion Center Davisville, RI, and Naval Shipyard Portsmouth, Kittery, ME. NCCOSC RDT&E Division Technical Document 2296.

NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 12

WASTE SAMPLING

12.1 PURPOSE. This chapter provides procedures for sampling **underground storage tanks (USTs)**, **drums**, **cylinders**, **Polychlorinated Biphenyl (PCB) transformers**, and other **hazardous** or **solid wastes** which pose a threat to human health and the environment. The main objective of waste sampling is to determine waste composition, verify location and extent of contamination (in order to protect human health and the environment in case of accidental release from the reference or known sources), or to meet regulatory monitoring requirements.

12.2 SCOPE. The scope of this chapter is to provide general procedural guidance to technical personnel who perform **waste sampling.** This waste sampling may be for site assessments or for meeting regulatory requirements mandating proper storage, handling, and disposal of materials in drums, cylinders, USTs, PCB transformers, oil spills, and other wastes at the site. It is intended to provide a framework for the development of site specific waste sampling procedures. For general sampling procedures and requirements refer to **Chapter 4** of this manual.

The following sampling subjects are included within the scope of this chapter:

- Underground Storage Tanks
- Surface sampling
- Polychlorinated Biphenyls
- Containers/Drums
- Oil Spills
- Waste

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. Contact responsible authority.

The main objectives of waste sampling are as follows:

- Provide relevant data for the protection of on-site and off-site personnel.
- Monitor groundwater quality at treatment, storage and disposal sites.
- Determine the presence of any USTs, drums, cylinders, PCB transformers, and other hazardous wastes which require special handling.
- Determine waste characteristic, toxicity and hazards present at the site due to reference or known sources.
- Determine alternative disposal criteria in accordance with all applicable Federal, state, and local regulations.
- Determine special procedural requirements for the handling and sampling of PCB contaminated articles, PCB containers, PCB contaminated transformers, and PCB transformers at the site.
- Evaluate and establish the **Spill Prevention Control and Counter-measure (SPCC) Plan** (along with proper health and safety procedures) as indicated by the specific circumstances, to ensure that oil spill sampling requirements are satisfied subsequent to an accidental release from the site.
- Define practices and procedures for safe handling of the drums and cylinders containing hazardous materials or wastes. The appropriate procedures for handling drums before sampling depends upon the content of the drums. The drums should be inspected before handling and sampling. In most compliance cases the materials contained in the drum or cylinder are known. Appropriate procedures for handling the suspected materials should be followed. However, depending on site control of the materials placed in drums or cylinders, additional safety practices should be considered.
- Safe handling of known materials. Unknown materials compatibility or sampling during remediation are included in the Navy Installation Restoration (IR) program.

NOTE:

Personnel should follow precautionary measures with unlabeled drums, cylinders, USTs, PCB transformers, and other wastes unless their contents are characterized or known.

12.2.1 Underground Storage Tanks (USTs). The scope of work for UST sampling provides information and guidance applicable to the regulation of USTs containing petroleum products and known stored chemical substances to be sampled at the site. If the sampling of 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 concern UST sampling:

- **CFR 112**: *EPA Regulation on Oil Pollution Prevention*
- **CFR 280**: *EPA Technical Standards and Corrective Action Requirements for Owners and Operators of USTs*

The following are included in the above references:

- When tank volume, including piping, is 10 percent or more beneath the ground surface
- A tank holding listed hazardous wastes or wastes meeting the Subtitle C criteria of the **Resource Conservation and Recovery Act (RCRA),** or a mixture of such wastes and other regulated substances
- The Federal regulation outlines 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 all listed USTs at the site and identify the regulatory requirements (including the compliance of each UST) for sampling, monitoring, and removal where necessary.

12.2.2 Polychlorinated Biphenyls (PCBs). The scope of work for PCB sampling provides information and guidance for PCB handling and sampling in accordance with applicable Federal, state, and local regulations.

The following references concern PCB sampling:

- **Toxic Substance Control Act (TSCA)**: *PCB requirements, as implemented by EPA regulations*
- **40 CFR 760-761**: *EPA regulations for controlling PCBs*

The following are included in the above references:

- Areas containing PCBs in regulated amounts are not open to the general public.
- Prior to sampling, all PCB transformers should 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 **Department of Transportation (DOT)** regulations.
- Non-leaking PCB articles and equipment, or leaking PCB items if they are placed in non-leaking containers with absorbent, may be put in temporary storage areas for up to 30 days.
- The site specific SPCC Plan must 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.

When performing PCB field sampling, proper health and safety protocol must be followed in accordance with **Occupational Safety and Health Administration (OSHA)** 29 CFR 1910.120. All items containing PCB, or unknown items related to PCB, must be considered potentially contaminated with PCB and handled according to the above mentioned applicable regulations.

12.2.3 Containers/Drums. The scope of work for container/drum sampling provides information and guidance applicable regulation of container/drum handling and sampling in accordance with Federal, state, and local regulations. The scope of work for container/drum sampling identifies a unique obstacle to sampling personnel such as drum staging, identification of the drums, and opening of the drums.

WARNING

The opening of drums is a major health and safety issue and proper precautionary measures must be taken while performing sampling at the site.

The scope of drum sampling includes the following:

- Accidents may occur during the opening of drums and other containerized hazardous materials at the site, such as detonation, explosion, fire, vapor generation, and physical injuries from handling of drums at the time of sampling.
- Applicable regulations are: OSHA 29 CFR 1910 and 1926, which includes general requirements and standards for storage, handling, and maintaining drums; 40 CFR 265; EPA requirements for types of containers and management of drums; and 49 CFR 171 through 178, DOT regulations that must be followed in field sampling.
- Prior to sampling drums, all drums should be visually inspected to gain as much information as possible about their content.
- Prior to drum sampling, a survey can be made to classify the drums into preliminary hazard categories. For example:
    - ° Radioactive
    - ° Leaking/deteriorated
    - ° Bulging
    - ° Explosive/shock sensitive
    - ° Lab waste

As a precautionary measure, all unknown drums should be handled and sampled with minimum Level B protection to characterize the content of the drums. The scope of work for sampling drums also includes and addresses unstacked drums and non organized drums at the site.

12.2.4 Oil Spill. The scope of work for oil spill sampling off-shore and ashore includes the methodology to collect samples due to 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 shore oil spills that are important to sample: the surface layer (0-15 cm), which reflects the accumulation of the oil due to offshore spill(s) from washout to the beach and long term deposition of the contaminated products several feet deep at the beach due to oil spills.

The scope of work for oil spill sampling includes the following references:

- **40 CFR 109**: *EPA Regulation on oil removal and contingency plan*
- **40 CFR 110**: *EPA Regulation on discharge of oil*
- **40 CFR 300**: *EPA Regulation on National Oil and Hazardous Substances Pollution Contingency Plan under Comprehensive Environmental Response, Compensation, Liability Act (CERCLA)*

The scope of work for oil spill sampling includes sampling the spill first. Since each spill and spill site is different, it is impossible to establish step by step guidelines for oil spill sampling. The best way to collect the spill sample is to collect the sample as soon as possible, gathering the greatest volume of oil before it spreads out or disperses. This also minimizes the effects of weathering alteration on the spilled oil. The scope of work for off-shore and ashore oil spill sampling must be in concurrence with the SPCC Plan, personnel **Health and Safety Plan (HASP)** (site specific) required to handle sampling events, which provides technical guidance associated with collection of samples.

12.2.5 Waste. The scope of work for waste sampling is to identify the waste present. It also involves characterization of the waste and how it adversely affects human health and environment on or in the vicinity. This scope also ultimately determines the disposal options available in accordance with Federal, state, and local regulations. Under RCRA (1976), an amendment to the Solid Waste Disposal Act to ensure that solid wastes are managed in an environmentally sound manner, there are two major distinct, yet interrelated, programs such as:

- Subtitle D, which promotes and encourages the environmentally sound management of solid waste
- Subtitle C, which promotes and encourages the environmentally sound management of hazardous waste

Hazardous wastes are those listed under RCRA 40 CFR 260, or exhibit any of four characteristics:

- Ignitability
- Corrosivity
- Reactivity
- Toxicity or TCLP (Toxic Characteristic Leaching Procedures)

The scope of work for waste sampling includes waste pile sampling, hazardous waste sampling, solid and debris waste sampling at the site. Sampling for the waste also includes contaminated solid materials such as sludges and powders.

12.2.6 Sewage Sludge. The scope of work for sewage sludge sampling is to collect and analyze representative samples of sewage sludge prior to the time it is applied to the land, placed on a surface disposal site, or fired in a sewage sludge incinerator. It involves characterization of the sewage sludge to determine by which method it can be disposed in accordance with Federal, state, and local regulations. While the CWA regulates beneficial use and disposal of sewage sludge, RCRA regulates disposal of non-hazardous sludge in a municipal landfill and land disposal of hazardous sludge.

The scope of work for sewage sludge sampling includes the following references:

- **40 CFR 258**: *Criteria for municipal solid waste landfills*
- **40 CFR 268**: *Land disposal restrictions*
- **40 CFR 503**: *EPA Standards for the use or disposal of sewage sludge*

The scope of work for sewage sludge sampling includes sampling the sewage sludge and domestic septage which will be disposed.

12.3 HAZARD AND SAFETY PRECAUTIONS.

See **Chapter 3, Section 3.3.3**.

12.3.1 Container Hazards.

WARNING

If drums or cylinders show signs of swelling or bulging, do not move or sample them until the pressure can be safely relieved.

Relieve the excess pressure prior to opening, if possible, from remote location using devices such as a pneumatic impact wrench or hydropic penetration device. If pressure must be relieved manually, place explosion resistant plastic barriers between the worker and drums/cylinders.

NOTE:

The integrity of the drums may dictate that overpacking is necessary prior to sampling. Therefore, overpack drums should be available.

Reseal the open bungs and drill openings as soon as possible with new bungs or plugs to avoid vapor release. If open drums can not be resealed then put them into the overpack drum.

Bulging drums are extremely hazardous. Wherever possible, do not move drums that may be under internal pressure, as evidenced by bulging or swelling.

Laboratory packs can be an ignition source for fires at hazardous waste sites. They sometimes contain shock sensitive materials. Consider such containers as explosive materials and use extreme care in opening these packs. Prior to opening lab packs, make sure all non-essential personnel are moved to a safe place. Whenever possible, use a grappler unit constructed for explosive containment for initial handling or sampling of such lab pack. Finally, maintain continuous communication with the Site Safety Officer until operations are complete.

12.3.2 USTs and Other Confined Spaces. For USTs and other confined spaces, the **Health and Safety Plan (HASP)** must be followed properly to ensure worker safety.

WARNING

The confined spaces entry must be followed properly with a stand-by unit on-site available at all times while performing confined space sampling in the UST.

The following steps should be taken before confined space entry takes place:

1. Ventilate thoroughly prior to entry.
2. Disconnect connecting pipelines.
3. Prior to entry, take air samples to prove the absence of flammable or other hazardous vapors and to demonstrate that an adequate level of oxygen is present.
4. Provide the entry team with proper respiratory protection, protective clothing, safety harnesses, and ropes.
5. Provide the stand-by team with the gear mentioned above for entry team.
6. Establish life line signals prior to entry so that field sampling personnel can communicate by tugging on the rope.
7. Maintain the ladder in accordance with OSHA regulations.

12.3.3 Explosive Hazards. Due to the inherent dangers in sampling explosives and potential or suspected explosives, only those individuals who have been trained and certified in the proper handling of these materials shall participate in sampling activities. See OPNAVINST 8023.2 for qualifications and certification requirements. Special considerations are required when sampling explosive wastes which may be 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. General safety considerations include wearing personal protective equipment such as flameproof clothing, caps , safety goggles or face shields, conductive shoes and respirators where applicable. 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 therefore imperative that sampling personnel have a thorough knowledge of the characteristic dangers and safety requirements for individual explosive materials. Information on explosives may be obtained from local explosive ordnance disposal (EOD) divisions or the Ordnance and Explosive Safety Office at NSWC Indian Head, MD. For explosive safety and handling requirements refer to NAVSEA OP5 Volume 1, “Ammunition and Explosives, Ashore Safety Regulations for Handling, Storing, Production, Renovation, and Shipping”.

WARNING

Only trained and certified explosive personnel shall handle, sample or test explosive or suspected explosive materials. Certified explosives experts can be contacted at facilities listed in Appendix G of this manual.

12.4 PREPARATION. In a case where the nature of the hazardous waste is unknown, such as an abandoned disposal site, sampling personnel should take additional precautions to protect themselves from direct contact with waste materials. For additional guidance refer to the Navy IR program. To characterize waste materials for the Resource Conservation and Recovery Act (RCRA) or to verify the contents in drums, cylinders, tanks, or transformers, it is important that representative samples be collected and analyzed.

WARNING

Sampler / sampling recommendations and strategies are presented in **Appendix E**.

12.4.1 Preparation for UST Sampling. Before starting UST sampling, follow the health and safety guidelines in **Section 12.3** of this chapter. Prior to UST sampling, tightness tests, if necessary, should be performed to assure that there is no leakage of the product from the UST and/or piping carrying the fluid. If it is necessary to enter into a tank or vault for sampling, strict procedures for confined space entry must be followed by qualified and experienced personnel.

12.4.2 Preparation for PCB Sampling. The toxic nature and degree of hazards posed by PCBs requires a high level of caution to be taken while sampling for PCBs. Appropriate protection gear and clothing should be worn by sampling and support personnel at the site during sampling events. Proper hand gloves must be used during sampling. Spill prevention and control must be planned in advance if transformer sampling is performed on site. Plastic pads and sorbent materials should be available at the site.

12.4.3 Preparation for Drum Sampling. The opening of closed drums prior to sampling entails considerable risk if not done with the proper techniques, tools, and safety equipment. Prior to sampling, all drums should be staged in easy access areas. Also, any standing water or other material should be removed from the top of the container so that the representative nature of the sample is not compromised when the container is opened. Also, there is the possibility of water reactive compounds being in the drums. Manual and remote drum sampling techniques are discussed below.

12.4.3.1 Manual Drum Opening.

12.4.3.1.1 Bung Wrench. The most common method for opening drums manually is the use of a universal bung wrench. These wrenches have fittings to remove nearly all commonly encountered bungs. They are usually constructed of cast iron, brass, or a bronze-beryllium non-sparking alloy formulated to reduce the likelihood of sparks. The use of the non-sparking wrench is highly recommended. However, the use of a non-sparking wrench does not completely eliminate the possibility of a spark being produced.

The fitting on the wrench matching the bung to be removed is inserted into the bung and the tool is turned counterclockwise to remove the bung. Proper protection and safety is necessary while opening drums.

If drums or cylinders show signs of swelling or bulging, do not move or sample them until the pressure can be safely relieved.

12.4.3.1.2 Drum Deheader. The drum can be opened manually when the bung is not removable with a bung wrench by using a drum deheader. This tool is constructed of forged steel with an alloy steel blade and is designed to cut the lid off a drum or part way off by means of 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. Moving the handle of the deheader up and down while sliding the deheader along the chime of a drum that has been damaged or badly dented, it may not be possible to cut the entire top of the drum. Since there is always the possibility that the drum is under pressure, cut the drum a little to release the pressure before cutting the whole lid off the drum. Proper health and safety procedures must be followed using a drum deheader.

12.4.3.2 Remote Opening.

WARNING

Before opening drum, ensure that the drum is in an area that has secondary containment.

12.4.3.2.1 Hydraulic Device. Recently, remotely operated hydraulic devices have been fabricated to open drums remotely. These devices use hydraulic pressure to pierce the wall of the drum. They consist of a manually operated pump which pressurizes oil through a length of hydraulic line. The metal point piercing device is attached to the end of this line and is pushed into the drum by hydraulic pressure.

The piercing device can be attached to the drum so that a hole for sampling can be made in either the side or the head/lid of the drum. Some of the piercers are hollow or tube-like so that they can be left in place, if desired, and serve as a permanent tap or sampling port. The piercer is designed in such a way that it establishes a tight seal after penetrating the container.

12.4.3.2.2 Pneumatic Devices. Pneumatic devices utilizing compressed air have been designed to remove drum bungs remotely. A pneumatic bung remover consists of a compressed air supply that is controlled by a heavy-duty, 2-stage regulator. A high pressure line of desirable length delivers compressed air to a pneumatic drill, which is adapted to turn a bung fitting selected to fit the bung to be removed. An adjustable bracketing system has been designed to position and align the pneumatic drill over the bung. The bracketing system must be attached to the drum before the drill can be operated.

12.4.4 Cylinder sampling. The compressed gas cylinder sampling and identification of unknown products pose a great environmental and personal risk and should only be performed by persons with appropriate training and experience. The local safety authority should be contacted prior to sampling.

12.4.5 Underground Storage Tanks (USTs), Vacuum Trucks, Process Vessels and Similar Large Containers. Prior to the initiation of the sampling event, all containers should be inventoried. All available information concerning each container should be recorded in the Field Log Book/Field Notes including:

- 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 an identification number for present and future reference. Enamel spray paint is often suitable for this purpose. Photographs of the numbered vessels can prove useful in documenting the containers' condition and can provide a permanent record.

The procedure used to open a large containment vessel to provide access to its contents will vary with different containers. Most large tanks and vacuum trucks will have valves near the bottom of the tank and hatches near the top. It is most desirable to collect samples from the top of a tank for several reasons. The integrity of valves near the bottom of the tank cannot be assured. The valve may be immobile or may break or become jammed in the open position resulting in the uncontrolled release of the tank's contents. Secondly the contents of a large vessel may become stratified. Collecting a sample from the bottom will not permit the collection of a sample of each strata. Instead a cross-sectional sample of the tank's contents should be obtained from the top access.

In opening and sampling larger containment vessels, precautions must be considered to assure personal health and safety. Accessing storage tanks requires a great deal of manual dexterity. It 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 must always perform the sampling; one to open the hatch and/or collect the actual 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. If necessary, the release valve should be opened slowly to bring the tank to atmospheric pressure. If the tank pressure is too great or venting releases gases or vapors, discontinue venting immediately. Measure releases to the atmosphere with portable field instrumentation and record in **Field Log Book/ Field Notes**.

If no release valve exists, slowly loosen hatch cover bolts to relieve pressure in the tank. Again, stop if pressure is too great or if a release occurs. Do not remove hatch cover bolts until tank is at atmospheric pressure.

If a discharge to ambient air occurs, sampling may need to be postponed until the proper equipment is available to control the release.

12.5 SAMPLE COLLECTION PROCEDURES.

12.5.1 Large Tanks. After the tank has been stabilized, sample collection may begin using one of the previously recommended samplers for containerized liquids and solids and employing the proper safety precautions and backup personnel. If the contents of the tank have stratified, each strata should be sampled discretely. At a minimum, a top, middle and bottom sample should be collected. If the container has separate compartments, each should be sampled separately at varying depths, as required.

Depending on the objective of the sampling event (e.g., characterization for disposal), compositing of samples in the laboratory on a weight/weight or volume/volume basis prior to analysis may be permissible. Knowledge of the process for filling the tank will assist with the decision to composite. Hot spots and separation of materials in compartments may be advantageous in reducing disposal costs where only a portion of the tank is contaminated. Compositing of the sample may result in low reported results from the laboratory that may not reflect overall concentration.
A well designed sampling plan is needed to ensure sampling and provides the data necessary for the decision.

Upon retrieval of the sample, immediately transfer the sample into the sample bottle.

12.5.2 Surficial Sampling. This recommended protocol outlines procedures and equipment for the collection of representative wipe, chip, sweep, and rinsate samples.

Surficial sampling is used to assess the existence and/or extent of contamination on various surfaces rather than in a soil, water, or air matrix. For example, the interior of a building may be assessed by collecting wipe samples of the process vessels and interiors of ventilation ducts. Though all four types of sampling are for similar purposes, these types of sampling are performed in very different ways because they are intended to assess different surface areas.

12.5.2.1 Wipe Samples. This method of monitoring surficial contamination is intended for non-volatile species of analytes (e.g., PCB, lead, Tetrachlorodibenzo-p-dioxin (TCDD) and Tetrachlorodibenzofuran (TCDF)) on non-porous surfaces (e.g., metal, glass). Sample points should be carefully chosen and should be based on site history, manufacturing processes, personal practices, obvious contamination, migration pathways and available surface area. Suggested sampling points include process vessels, ventilation ducts and fans, exposed beams, window panes, etc. The area wiped should be large enough to provide a sufficient amount of sample for analysis (smaller sample volumes cause higher detection limits).

To collect a wipe sample, the following equipment is needed:

- Ruler or measuring tape to measure out the area being wiped
- Disposable surgical gloves, to be changed prior to handling clean gauze, sample container or solvent
- Gauze pad inside a precleaned container

To facilitate the collection of a wipe sample, 3 in. x 3 in. gauze should be utilized. The use of filter paper for wipe sampling is not recommended. Filter paper will tend to rip and crumble if the surface wiped is slightly rough. If filter paper is to be used, it should be four inch diameter heavy gauge paper, such as Whatman #4 Filter Paper.

Once the sample location has been determined, sample collection can begin. For each sample, a new pair of disposable gloves should be worn to avoid contamination of the collected samples (i.e., natural body oils from hands). The following is for wipe sample procedures:

1. It is recommended that an area be premeasured (e.g., 25 cm x 25 cm) to allow for easier calculation of final results. However, this is not always feasible and may be done after the area is wiped.
2. Wearing a new pair of disposable surgical gloves, remove the gauze pad from its sterile wrapping container and soak it with the appropriate solvent.
3. Wipe entire area to be sampled once in the horizontal direction and once in the vertical direction, applying moderate pressure.
4. Wipe the entire area so that all the sample material is picked up.
5. Place the gauze pad into 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 in order to provide additional data for interpretation. The field blank will help to identify potential introduction of contaminants from the pad, solvent, sample container, or ambient air conditions. 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 bottle.

In some cases, the laboratory should be told to rinse the sample jars with the appropriate extraction or digestion solvent (depending on the analysis to be performed) when transferring sample to the extraction glassware. This will ensure that the entire sample has been removed from the container.

12.5.2.2 <u>Chip Samples</u>. This method of monitoring surficial contamination is intended for non-volatile species of analytes (e.g., PCB, TCDD and TCDF) on porous surfaces (e.g., cement, brick, wood). Sample points should be carefully chosen and should be 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, loading dock areas, etc. The sampling area should be large enough to provide a sufficient amount of sample for analysis (smaller sample volumes cause higher detection limits). To facilitate the calculations once the analytical data is received, the area sampled should be measured. To collect a chip sample, the following equipment is needed:

☐ Ruler or measuring tape to measure out the area to be sampled
☐ Disposable surgical gloves, to be changed prior to collection of each sample
☐ Decontaminated chisel of borosilicate construction and hammer or electric hammer
☐ Natural bristle brush and a dust pan lined with aluminum foil or one that is dedicated, decontaminated and constructed of a pre-approved material which will not interfere with the contaminants of concern
☐ Container for sample

Once the sample location has been determined and marked off, sample collection can begin. For each sample, a new pair of disposable gloves should be worn to avoid contamination of the collected samples (i.e., natural body oils from hands). The following is for chip sample procedures:

1. Break up the surface to be sampled using a decontaminated chisel and hammer.
2. An effort should be made to avoid scattering pieces out of the sampling boundary. Any pieces that fall outside the sampling area should not be used.
3. The area should be chipped to less than one-quarter inch (preferably 1/8 in.).
4. Record how deep chips were taken and write in Field Log Book/Field Notes.
5. Collect the chipped pieces using a dedicated, decontaminated dust pan and natural bristle brush and transfer the sample directly into the bottle.

12.5.2.3 <u>Sweep Samples</u>. This method of monitoring surficial contamination is intended for non-volatile species of analytes (e.g., PCB, TCDD and TCDF) in residue found in porous (e.g., asphalt) or non-porous (e.g., metal) surfaces. Sweep sampling allows the collection of dust/residue samples that may help in the assessment of contaminant determination and delineation. Sample points should be carefully chosen and 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), street gutters where contaminated sediments may have migrated and accumulated, etc. The area sampled should be large enough to provide a sufficient amount of sample for analysis.

WARNING

To collect a sweep sample, the following equipment is needed:

- ☐ 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 preapproved material which will not interfere with the contaminants of concern
- ☐ Disposable surgical gloves to be changed prior to collection of each sample
- ☐ Container for sample

Once the sample location has been determined, sample collection can begin. Wearing a new pair of disposable gloves, sweep all residue in the area to be sampled onto a decontaminated or dedicated dust pan or directly into the sample container. A decontaminated or dedicated spatula may be used to aid in transferring the sample into the sample bottle.

12.5.2.4 Rinsate Samples. This method of sampling is utilized to determine if surfaces contain hazardous waste residue after being cleaned. It is normally associated with drum storage pads, floors of buildings, and the inside of waste tanks.

Rinsate samples are performed by collecting the water from the last rinse when cleaning a tank or surface area. This water is then analyzed for the known chemical contaminants and compared against a blank consisting of the same type of water.

12.5.3 Transformers. The peculiarities that are associated with transformers warrant that these containers be considered separate from drums and tanks. Because transformers are often located in secured, out-of-the-way locations, access may present a problem. For pole mounted transformers, a power operated scissor lift or cherry picker may be needed. In other cases, the transformer may be in an underground cell.

The toxic nature and degree of hazard posed by PCBs which may be present in a transformer dictate that a high level of caution be used. Appropriate protection should be worn by sampling and support personnel. Spill prevention and control must be planned; plastic sheeting and absorbent pads should be employed.

> **Most importantly, the transformer must be certified as "off-line" and de-energized by an electrician or other responsible person. Also, relieve internal pressure before opening the transformer.**

Once the power source to the transformer is cut and spill control measures (plastic sheeting on ground and/or floor surface of lift) are in place, the cover of the transformer can be removed with hand tools. A sample of the dielectric fluid is most efficiently obtained with a disposable glass **Composite Liquid Waste Sampler (COLIWASA)** (See **Section 12.5.4.2**).

In order to obtain a representative sample, the following procedure should be used:

1. Lower the COLIWASA at a rate that allows the levels of the fluid, both inside and outside the sampler, to remain the same.
2. When the sampler reaches the bottom of the transformer, close it and as it is retrieved, wipe the COLIWASA with a disposable absorbent pad.
3. Transfer the sample directly into the sample bottle. 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 materials. However this should only be done after consultation with the 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:**
> The transformer drain valve should never be utilized for sample collection for several reasons. 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 the transformer's contents. Secondly, it is likely that transformer contents may have stratified. Since PCBs are heavier than other insulating oils, this stratification may prevent the collection of representative sample. Samples obtained from the valve near the bottom of the transformer might reveal higher PCB concentrations than the true concentration of the total dielectric fluid

12.5.4 Containerized/Drum Sampling. Some of the most difficult liquids to sample are those stored in a container. Several factors play an important role in determining the sampling method to be used. These include the location of the container, the location and size of the opening on the container, and the type of equipment that is available for sampling. The health and safety of sampling personnel also plays a key role in determining which sampling method should be used.

No matter what type of sampler is chosen, it must be utilized in such a manner that allows collection of all phases present in the container. Rarely does a container hold a homogeneous mixture of material.

Sampling devices for containerized liquids and their procedures for use are presented below. Other sampling devices, which may be considered appropriate, include the Bacon Bomb, Kemmerer Depth, and a Weighted Bottle sampler (See **Section 7.5.1** of this manual). Additional updated references on sampling containerized liquids are found in ASTM Volume 14.03 for waste sampling. Material sampling of oils, paints and other containerized chemicals are found in ASTM volumes relating to these materials.

Sampling containerized materials presents unique obstacle to sampling personnel, whether the container involved is a fiber drum or vacuum truck. Container staging, identification, and opening are all issues to be considered. Health and safety precautions associated with sampling containerized materials are generally more stringent. **Quality Assurance (QA)** guidelines for waste samples, as opposed to environmental samples, are unique; therefore, each site should be considered individually. When sampling waste materials, high levels of contaminants can be expected. Therefore, trip and field blanks may be inappropriate. However, if residual or low-level waste/chemicals are expected (e.g., sampling contaminated soils in drums or containers) trip and field blanks may be appropriate.

Prior to the initiation of the sampling event, all containers should be inventoried. All available information concerning each container should be recorded in the Field Log Book/Field Notes including the:

- Type of container
- Total capacity estimate
- Actual capacity (if container is open)
- Markings, labels, color, origin, condition, etc.

Photographs **may** be taken to provide a permanent record.

Depending on the location and position of the containers, it may be necessary to upright and/or relocate them prior to sampling.

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 safely relieved.

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.

The sampling of tanks, containers, and drums present unique problems not associated with natural water bodies. Containers of this sort are generally closed except for small access ports, manways, or hatches on the larger vessels or taps and bungs on smaller drums. The physical size, shape, construction material, and location of access will limit the types of equipment and methods of collection.

When liquids are contained in sealed vessels, gas vapor pressures build up, sludges settle 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. Preliminary sampling of any headspace gases may be warranted. As a minimum, a preliminary check with an explosimeter or an organic vapor analyzer will determine levels of personnel protection and may be of aid in selecting a sampling method.

In most cases it is impossible to observe the contents of these sealed or partially sealed vessels. Since some layering or stratification is likely in any solution left undisturbed over time, a sample must be taken that represents the entire depth of the vessel.

Agitation to disrupt the layers and re-homogenize the sample is physically difficult and almost always undesirable. In vessels greater than 1 meter in depth the method of choice is to slowly, in known increments of length, lower the suction line from a peristaltic pump. Discrete samples can be collected from various depths then combined or analyzed separately. If the specific depth of the sample is important, a discrete sample such as the Kemmerer type may be required. In situations where the reactive nature of the contents are known, a small submersible pump may be used.

When sampling a previously sealed vessel, a check should be made for the presence of a bottom sludge. This is easily accomplished by measuring the depth to apparent bottom then comparing it to the known interior depth.

Liquid samples from opened containers (55-gallon drums) are collected using lengths of glass tubing (Drum Thief). The glass tubes are normally 122 cm in length and 6 to 16 mm inside diameter. Larger diameter tubes may be used for more viscous fluids if sampling with the small diameter tube is not adequate. This method should not be attempted with less than a two-man sampling team and protective gear should be worn.

Use of a Drum Thief is simple, relatively inexpensive, quick and collects a sample without having to decontaminate equipment. It does, however, have serious drawbacks. Most low density fluids do not hold well in the glass tubes. A great deal 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. These include the incorporation of a small suction device (i.e., 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 then drawn to form a much smaller orifice. This allows the use of larger diameter tubing; therefore, a greater volume of sample per attempt, while reducing the material loss from the tube bottom normally associated with larger diameter tubes. The COLIWASA is a much cited sampler designed to permit representative sampling of multiphase wastes from drums and other containerized wastes. The sampler is commercially available or can be easily fabricated from a variety of materials including PVC, glass, or Teflon®. In its usual configuration it consists of a 152 cm by 4 cm (inside diameter) section of tubing 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 raising and lowering the neoprene stopper. A current recommended model of the COLIWASA is shown in **Figure 12-1**, however, the design can be modified and/or adapted somewhat to meet the needs of the sampler.

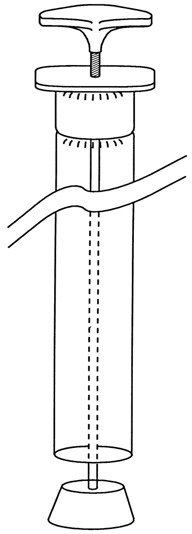


Figure 12-1 COLIWASA Sampler

The COLIWASA is primarily used to sample most containerized liquids. The PVC COLIWASA is reported to be able to sample most containerized liquid wastes except for those containing ketone, nitrobenzene, dimethylforamide, mesityloxide and tetrahydrofuran. A glass COLIWASA is able to handle all wastes that are otherwise not able to be sampled with the plastic unit except strong alkali and hydrofluoric acid solutions. Due to the unknown nature of most containerized waste, it would therefore, be advisable to eliminate the use of PVC materials and use samplers composed of glass or Teflon®.

The major drawbacks associated with using a COLIWASA concern decontamination and costs.

NOTE:

Disposable COLIWASAs are commercially available and are recommended.

It should be noted that in some instances disposal of the tube by breaking it into the drum may interfere with eventual plans for the removal of its contents. The use of this technique should, therefore, be cleared with the project officer or other disposal techniques evaluated. Heat shrinkage Teflon® tubing or other types of Teflon® coating can also be used to cover the stainless steel rod if contact of the stainless steel with the waste is undesirable.

12.5.4.1 Procedures for Drum Sampling Using Glass Tubes (Drum Thief).

1. Record the drum identification number or label. Drum should be labeled to ensure the identity of

drum for later confirmation sampling or disposal. Open drum to be sampled.

2. Remove cover from sample container.
3. Lower glass tubing to the point that it almost touches the bottom of the container. Try to keep at least 30 cm of tubing above the top of the container.
4. Allow the waste in the drum to reach its natural level in the tube.
5. Cap the top of the tube with a safety-gloved thumb or a rubber stopper.
6. Carefully remove the capped tube from the drum and insert the uncapped end in the sample container.
7. Release the thumb or stopper on the tube and allow the sample container to fill to approximately 90 percent of its capacity or no headspace for volatiles.
8. Repeat steps 2 through 7 if more volume is needed to fill the sample container.
9. Remove the tube from the sample container and replace the tube in the drum.
10. Cap the sample container tightly with a Teflon®-lined cap and affix the sample identification tag.

NOTE:

See the initial discussion in this section for exceptions.

11. Replace the bung or place plastic over the drum.
12. Place sample container in a resealable plastic bag (one per bag). Place bag in paint can for transport.
13. Mark the sample identification number on the outside of each paint can and complete **Chain-of-Custody Record** and **Field Log Book/Field Notes**.

Optional Method (if sample of bottom sludge is desired):

1. Remove cover from container opening and sample container.
2. Lower glass tubing to the point that it almost touches the bottom of the container. Try to keep at least 30 cm of tubing above the top of the container.
3. Allow the waste in the drum to reach its natural level in the tube.
4. Gently push the tube towards the bottom of the drum into the sludge layer. Do not force it.
5. Cap the top of the tube with a safely-gloved thumb or rubber stopper.
6. Carefully remove the capped tube from the drum and insert the uncapped end in the sample container.
7. Release the thumb or stopper on the tube and allow the sample container to fill to approximately 90 percent of its capacity. 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 if more volume is needed to fill sample container and recap the tube.
9. Proceed as in steps 9 through 13 above.

NOTE:

1. If a reaction is observed when the glass tube is inserted (violent agitation, smoke, light, etc.) the sampling personnel should leave the area immediately.
2. If the glass tube becomes cloudy or smokey after insertion into the drum, the presence of hydrofluoric acid is indicated and a comparable length of rigid plastic tubing should be used to collect the sample.
3. When a solid is encountered in a drum (either layer or bottom sludge) the optional method described above may be used to collect a core of the material, or the material may be collected with a disposable scoop attached to a length of wooden or plastic rod.

Sources: *ASTM E-300-73 Standard Recommended Practices for Sampling Industrial Chemicals*, *EPA Technical Methods for Investigating Sites Containing Hazardous Substances*, *Technical Monograph I-29*, *Draft*, Ecology and the Environment, June 1981.

12.5.4.2 <u>COLIWASA Use Procedures</u>. Procedures can be adapted for use with other versions of COLIWASA samplers.

1. Choose the material to be used to fabricate the COLIWASA and assemble the sampler.
2. Make sure that the sampler is clean.
3. Check to make sure the sampler is functioning properly. Adjust the locking mechanism if necessary to make sure the neoprene rubber stopper provides a tight closure.
4. Wear necessary protective clothing and gear and observe required sampling precautions.
5. Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.
6. Slowly lower the sampler into the liquid waste. (Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same. If the level of the liquid in the sample tube is lower than that outside the sampler,

the sampling rate is too fast and will result in a non-representative sample).

7. When the sampler stopper hits the bottom of the waste container, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the closed position by turning the T handle until it is upright and one end rests tightly on the locking block.
8. 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.
9. Carefully discharge the sample into a suitable sample container by slowly pulling the lower end of the T handle away from the locking block while the lower end of the sampler is positioned in a sample container.
10. Cap the sample container with a Teflon®-lined cap, attach label and seal, record in Field Log Book/Field Notes, and complete sample analysis request sheet and **Chain-of-Custody** Record.
11. Unscrew the T handle of the sampler and disengage the locking block. Clean 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.

Sources: deVera, E.R., Simmons, B.P., Stephens, R.D., and Storm, D.L. *Samplers and Sampling Procedures for Hazardous Waste Streams*, EPA 6../2-80-018, January 1980.

12.5.5 Containerized Solids. The sampling of containerized solid materials (sludges, granulars, powders) is generally accomplished through the use of one of the following samplers:

- Scoop or trowel
- Waste pile sampler
- Veihmeyer sampler
- Grain sampler

Once the container to be sampled is opened, insert the decontaminated sampling device into the center of the material to be sampled, where possible. Retrieve the sample and immediately transfer it into the sampling bottle. When using the same sampling device, decontaminate the device thoroughly before collecting the next sample. Each sample should be sampled discretely. Depending on the objective of the sampling event (e.g., characterization for disposal) compositing of samples in the laboratory on a weight/weight basis prior to analysis may be permissible.

12.5.6 Waste Pile Sampling. This recommended protocol outlines general procedures for collecting samples from waste piles and other waste materials, equipment necessary for sampling, and the adequate representation of the material. Also presented will be necessary factors for consideration when formulating a sampling plan. Because of the variables involved in waste material sampling, including: shape and size of piles; size, compactness and structure of the waste material; and make-up throughout the material, exact procedures cannot be outlined for every sampling situation. Considerations must be made for the above mentioned variables, the purpose of sampling, and the intended use of the data to help determine correct sampling methodology.

12.5.6.1 Considerations for the Sampling Plan. The physical and chemical make-up of the waste pile and the purpose of sampling should be considered in planning for the sampling. Information about these items is presented below.

12.5.6.2 Shape and Size. Shape and size of waste material and waste piles may vary greatly in a real extent and height. The pile may be cone shaped, long and rectangular, square, oval or irregularly shaped. State and Federal regulations often require a specified number of samples per volume of waste, therefore, size and shape may be used to calculate volume and to plan for the correct number of samples. Shape must also be considered when planning physical access to the sampling point and the type of equipment necessary to successfully collect the sample at that location.

12.5.6.3 Sampling Procedures. Sampling devices most commonly used for small waste piles are 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 12-2**). The outer tube has a conical pointed tip that permits the sampler to penetrate the material being sampled. The inner tube is rotated to open and close the sampler. A thief is used to sample dry granules or powdered wastes whose particle diameter is less than one-third the width of the slots.

Figure 12-2 Thief Sampler

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 12-3**). A trier samples moist or sticky solids with a particle diameter less than one-half of it's diameter. Triers 61 to 100 cm long and 1.27 to 2.54 cm in diameter are available. A larger trier can be fabricated.

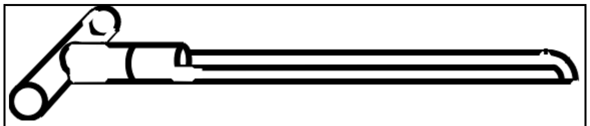


Figure 12-3 Sampling Trier

12.5.6.4 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. A composite sample composed of five individual samples taken with a shovel, six inches beneath the surface, should represent a sampling point. Depending upon the size of the waste pile, three, five, or seven such composite sampling points should be representative of the waste pile. A thief or trier sampler would provide a more representative sample compared to a shovel, if such is a practical substitution for the shovel.

When the waste pile is composed of heterogeneous material, an effort should be made to determine the source, nature, and extent of the deposited waste. If it is possible to sample the waste sources contributing to the waste pile, the waste characteristics could be easily obtained. If it is determined that any of the waste within a heterogeneous waste pile is a hazardous waste, the entire waste pile is a hazardous waste by definition, and should be managed as such, unless the waste pile can be segregated into hazardous and non-hazardous components.

Waste piles containing unknown waste can be sampled using a three-dimensional random sampling strategy where the pile is divided into a three-dimensional grid system, the grids sections assigned numbers, and the sampling points then chosen using random-number tables or random-number generators. This strategy can be employed only if all points within the pile can be accessed. In completing the TCLP tests, the first one that indicates the material to be a hazardous waste should be sufficient ground to cease further testing on the waste pile. If any part of the waste pile is a hazardous waste, the entire pile becomes regulated in such a manner.

12.5.6.5 Composite Samples. Compositing of a number of individually collected samples in the field may be used to reduce analytical cost. The disadvantage of sample compositing is a loss of concentration variance data, whereas the advantage is a more representative sample from the universe that is being sampled. This sample collection technique is especially valuable when the waste pile is believed to be homogeneous. When a waste pile is believed not to be homogenous, individual random sampling is the preferred sampling technique. Compositing requires that equal amounts of material from each of the number of samples to be composited be combined and sufficiently mixed so that an aliquot sample can be removed for laboratory analysis. If the concentration of a regulated material exceeds a regulated level, the entire waste pile must be managed as regulated waste, or additional sampling and analyses must be performed to define regulated and non-regulated material.

To avoid the necessity of resampling in the event that a composited sample exceeds the regulated limit, it is essential to collect a sufficient volume of each initial sample so that sufficient sample will remain following compositing to allow for a different compositing scheme or even for the analysis of the component samples themselves. This requires that the initial field samples be retained until the laboratory results on the composited sample become available.

12.5.7 Sampling for Lead in Paint. X-ray fluorescence and direct laboratory testing may be used to determine the lead content of paint. However, X-ray fluorescence is a screening test because some interferences may make it unreliable. Laboratory testing should be used to determine the concentration of lead in solid materials.

To collect a paint sample suitable for lead determination, use a sharp knife to cut through the paint. Lift off 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 field log book\field notes.

A sample of about 2 inches by 2 inches is required for a determination. 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.

As a minimum, a paint sample should be collected from the door, window and wall of each room in the building being investigated. 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. As 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 percent by laboratory testing or greater than 1.0 mg/cm^2 by X-ray fluorescence. When the first sample from a door, window, or wall results in a lead content exceeding 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 about 1980 would not be expected to contain paint with elevated lead concentrations.

12.5.8 Ashore Oil Spill Sampling. The methods outlined in this section are for the collection of surface ashore samples whenever the spill occurs from the site or vessel. In this case, use of a suction device can be made to collect samples. The statistical design of the sampling should be the same no matter what devices are used to collect samples. Ashore oil spill samples can be collected using the same methodology used for offshore sampling, such as inverting the 4 fluid oz. jar and collect the liquid oil sample. Tar balls can be sampled for ashore sampling as mentioned in offshore sampling. Shallow pollution samples can be collected by using one of the following methods:

Soil Punch Sampling. This is a thin wall tube, 15 to 20 cm long, used to extract short cores from contaminated locations at beaches. The following steps are used to collect samples with this equipment:

1. Drive the tube into the potentially contaminated location with a wooden mallet.
2. Extract the tube and core.
3. Push out collected materials in mixing bowl, collect sample into label jar, store the sample at 4°C.
4. Fill out **Chain-of-Custody (COC)** Record.

Scoop or Shovel Sampling. Surface samples due to an ashore oil spill are collected with a scoop and shovel. The small plastic disposable scoop can used to collect representative samples. In this case proper Quality Assurance/Quality Control (QA/QC) procedures should be followed to ensure cross-contamination cannot occur during sampling events. Larger representative samples can be collected with the help of a shovel at the site.

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. Force the Hand Auger to the location where the sample is to be collected.
2. Auger should be twisted or screwed, extract the sample, pull the auger, and retrieve the sample in the jar.
3. Follow step 3 of the soil punch sampling procedure.

NOTE:

Power Augers can be used to collect samples. The procedure is the same as the Hand Auger.

12.6 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC). Appropriate protocols should be followed to ensure the accuracy of data collected during sampling (for additional information, see **Chapter 4, section 4.7**).

A QA/QC plan involves the establishment of a sampling protocol, which is designed to minimize source of error in each and every stage of the process, from sample collection to analysis and reporting of analytical data. Key elements are as follows (see **Chapter 3, Section 3.3.1** for Quality Assurance Plan elements):

- 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, which allows collection of subsurface samples.
- Use of a sampling device that minimizes disturbance of the chemistry of the media formation.
- Use of field decontamination procedures to minimize cross-contamination during sampling.

Collection of QA/QC samples and following **Chain-of-Custody** procedures.

12.7 SAMPLING EQUIPMENT LIST. Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to waste sampling:

- ☐ Safety equipment, as required
- ☐ Manual Drum Opening
  - ☐ Bung Wrench
  - ☐ Drum Deheader
- ☐ Remote Opening
  - ☐ Hydraulic Device
  - ☐ Pneumatic Devices
- ☐ Ashore Oil Spill Sampling

  - ☐ 4 fluid oz. jar
- ☐ Shallow pollution sampling
  - ☐ Sampling with a soil punch
  - ☐ Scoop or Shovel
  - ☐ Hand Augers or Power Auger
- ☐ Surficial Sampling
  - ☐ Wipe Samples
  - ☐ Ruler or measuring tape
  - ☐ Disposable surgical gloves
  - ☐ Gauze pad inside a pre-cleaned container
  - ☐ Four inch diameter heavy gauge paper, such as Whatman #4 Filter Paper
  - ☐ Disposable surgical gloves
- ☐ Chip Sampling
  - ☐ Ruler or measuring tape to measure out the area to be sampled
  - ☐ Disposable surgical gloves, to be changed prior to collection of each sample
  - ☐ Decontaminated chisel of borosilicate construction and hammer 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 which will not interfere with the contaminants of concern
  - ☐ Container for sample
- ☐ 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 preapproved material which will not interfere with the contaminants of concern
  - ☐ Disposable surgical gloves to be changed prior to collection of each sample
  - ☐ Container for sample
- ☐ Transformers
  - ☐ Disposable glass Composite Liquid Waste Sampler COLIWASA
- ☐ Containerized/Drum Sampling
  - ☐ Bacon Bomb
  - ☐ Kemmerer Depth
  - ☐ Weighted Bottle sampler
  - ☐ Drum thief (Glass Tubes)
  - ☐ COLIWASA
  - ☐ Disposable scoop attached to a length of wooden or plastic rod.
- ☐ Containerized Solids Sampling
  - ☐ Scoop or trowel
  - ☐ Waste pile sampler
  - ☐ Veihmeyer sampler
  - ☐ Grain sampler
- ☐ Waste Pile Sampling
  - ☐ Thieves
  - ☐ Triers
  - ☐ Shovels
- ☐ Sampling for Lead in Paint
  - ☐ X-ray fluorescence
  - ☐ Sharp knife to cut through the paint
  - ☐ Clean putty knife
  - ☐ Resealable plastic bag or appropriate container

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CHAPTER 13

CLEAN SAMPLING FOR AQUEOUS TRACE METALS AND TRACE ORGANICS

13.1 PURPOSE. The purpose of this chapter is to provide practical instructions on when and how to successfully use clean sampling procedures. Clean and ultraclean sampling procedures are specialized techniques needed to collect contamination-free water samples for low level trace metals and trace organic analysis. These procedures represent an increased level of effort (and cost) over established water sampling techniques (See **Chapter 7**) and are required to accurately measure the low, background concentrations of trace metals and organics present in natural waters (i.e. low parts per billion (ppb) to low parts per trillion (ppt)). Clean procedures are also needed to filter water samples without contamination for dissolved trace metals and organics where low detection limits are needed. At these low levels even "invisible" amounts of contamination from the atmosphere or surfaces contacting the sample can result in false high concentration data. Consequently, the guiding principle in clean chemistry is to avoid contamination during all steps in the sampling process (i.e. collection, processing, and analysis) and to document the lack of contamination through the liberal use of equipment, field and laboratory blank samples. Avoiding contamination is accomplished by using specially cleaned sampling equipment and exercising additional care during the entire sampling and analysis process.

13.2 SCOPE. Clean chemistry is a general term which encompasses all procedures needed to accurately measure the low (background) concentrations of trace metals and organics found in natural waters. Clean chemistry includes three separate components:

- Collection of water samples without contamination (clean sampling)
- Very sensitive analytical techniques capable of measuring trace metals and organics in water at the part per trillion level and below (clean analysis)
- Comprehensive quality assurance (QA) procedures to verify the lack of contamination during both field sampling and laboratory analysis

All three components must be successfully completed to produce accurate and defensible data. This chapter describes the clean sampling procedures required to collect water samples without contamination and the associated QA procedures needed to confirm a contamination-free sample.Very sensitive analytical techniques and their associated QA procedures are an essential component of clean chemistry but are beyond the scope of this chapter. However, it must be noted that even if a sample is collected cleanly in the field, inaccurate data can easily be produced during the laboratory phase. For example, contamination from the laboratory environment can occur during sample preparation (e.g. preservation, digestion, etc.) or analysis. Also, it is difficult to analyze samples at concentrations less than 1-10 ppb and inaccurate data can arise if the analyst does not deal effectively with interferences caused by chemicals (matrix) in the sample or with small signals caused by working near the detection limit of the analytical instrument used. This is especially true for trace metals in seawater samples, which must be analyzed using sophisticated extraction or preconcentration techniques to produce accurate data. The U.S. Environmental Protection Agency (EPA) is in the process of issuing new, clean chemistry (1600 series) analytical methods for both trace metals and trace organics which are specifically directed at making measurements in the low ppb to ppt range.

The clean sampling procedures discussed in this chapter are only applicable to the collection of natural waters and waste waters where low detection limit or dissolved trace metals or organics measurements are needed. Ambient (background) concentrations of these metals and organics in water are so low that even minute contamination can invalidate the sample. Clean sampling procedures are generally inappropriate (i.e. overkill) for total recoverable determinations of metals and organics in treated and untreated industrial effluents where concentrations are in the mid to high ppb range. The same is true for collecting biological (tissue) and geological (sediment) materials where the concentrations of these constituents can be even higher (parts per million (ppm) range). However, the guidance on eliminating or minimizing contamination in this chapter can be useful in improving the collection of other sample types including: organisms, soils, sludges, solid and hazardous wastes, and air-borne metals samples, whether particulate or volatile.

Clean sampling procedures for trace metals and organics are covered separately in this chapter. However, since the goal for clean sampling of both metals and organics is to avoid all external contamination, there is much in common between each set of procedures. The first three sections of this chapter give background and introductory information about clean sampling procedures. This information will provide sampling personnel an understanding of the fundamental concepts upon which clean sampling is based and will allow implementation of these procedures in an efficient and cost-effective manner. The introductory sections include:

- Background
- When to Use Clean Sampling Procedures
- Specialized Facilities and Equipment
- Hazards and Safety Precautions

The remaining chapter sections are organized by tasks needed to carry out a successful field sampling program using clean sampling procedures. All necessary QA procedures and samples are incorporated into these sections which include:

- Preparation for Field Sampling
- Field Sampling Procedures
- Post-Sampling Procedures

13.3 BACKGROUND. The need for low detection limit, clean chemistry procedures is a result of the EPA's increased emphasis in recent years on water-quality criteria (WQC) based permitting. Currently, compliance monitoring for trace metals is based on technology-based effluent limits which are in the mid to high ppb range. To accurately measure these technology-based limits for compliance monitoring and to avoid false positives, analytical methods with method detection limits (MDL) approximately one-tenth of the effluent limits are needed (See **Table 13-1**).These methods are routinely available in environmental laboratories and the reporting limits (i.e. minimum level (ML), ML= 3.18 times the MDL) for these methods are listed in **Table 13-1**. However, as the EPA changes to WQC-based effluent limits, more sensitive analytical techniques will be needed. The lowest freshwater and saltwater WQC are generally in the low ppb range. To accurately perform compliance monitoring, analytical techniques with MDL's ten times lower than the WQC will be required. The EPA has already issued several new, more sensitive methods (1600 series) to measure trace metals in water at WQC levels.

A second development which makes the use of clean chemistry procedures necessary, is the EPA's change from total recoverable to dissolved trace metal concentrations for compliance monitoring. This change was made because the dissolved metals more closely approximates the bioavailable (toxic) fraction of metal in the water column than total recoverable metal does. Dissolved metal samples are produced by filtering the raw water through a 0.45 :m filter in the field. This filtration step is highly prone to contamination and requires clean sampling procedures to produce valid data. **Figure 13-1** illustrates the relationship among various trace metal fractions (species) in water samples.

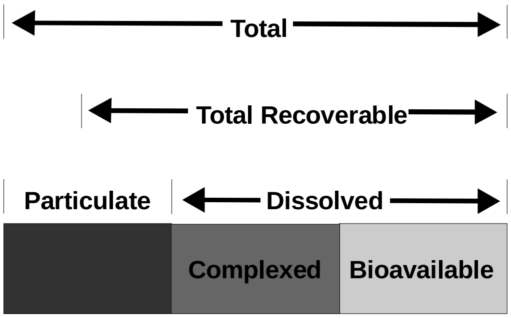

Figure 13-1 Physical Specification of Trace Metals in Water

13.3.1 **Trace Metals.** The ease of contaminating water samples with the trace metals and interfering substances cannot be overemphasized. The reason for this situation is two-fold. First, trace metals are everywhere in the environment, so contamination of water samples with external metals can occur during every step of the sampling and analysis process. Second, the natural (background) concentrations of trace metals in water as well as certain WQC are in the low ppb to ppt range (See **Table 13-1** Columns 5 and 7). These levels are so low that even the smallest piece of material can result in an erroneously high concentration value. As a result, avoiding sample contamination is the overriding goal of clean sampling procedures, and the approach is straightforward to ensure that any object or substance that contacts the sample is non-metallic and free from any material that may contain metals. When this goal is understood, most clean procedures are easy to understand as more or less common sense ways to keep water samples from coming into contact with potential external contaminants.

There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination during sampling include:

Table 13-1 Comparison of U.S. EPA Technology-Based and Water Quality Criteria (WQC) Based Minimum (Reporting) Levels (ML) and WQC with Typical Dissolved Metal Concentrations in Freshwater and Saltwater for 13 Priority Pollutant Metals

Concentration (Fg/L = ppb)

1 Element	2 EPA Technology-Based ML	3 Lowest EPA WQC-Based ML	4 Lowest EPA Freshwater WQC	5 Typical Dissolved Freshwater	6 Lowest EPA Saltwater WQC	7 Typical Dissolved Saltwater
Antimony	20.0	0.02	---	1.0	---	0.15
Arsenic	10.0	0.005	181.0	1.7	34.2	1.8
Beryllium	---	---	---	---	---	0.000
Cadmium	5.0	0.01	0.32	0.02	7.9	2
Chromium	20.0	0.5	10.5	1.0*	47.5	0.081
(VI)	25.0	0.1	3.1	1.5	2.5	0.21*
Copper	5.0	0.02	0.14	0.1	2.1	0.26
Lead	0.2	0.0002	0.012	0.004	0.025	0.002
Mercury	40.0	0.1	42.0	0.5	7.1	0.001
Nickel	5.0	2.0	5.0	0.08	71.0	0.48
Selenium	10.0	0.1	0.31	0.02	2.0	0.14
Silver	10.0	0.02	---	---	---	0.003
Thallium	20.0	0.5	28.0	20.0	73.0	0.013
Zinc						0.4

1 Technology-based minimum (quantitation) level = 3.18* method detection limit (MDL).
2. WQC-based ML from new EPA series 1600 methods.
3. Lowest EPA WQC for freshwater usually chronic total dissolved at 25 mg/L $CaCO_3$.
4. Typical dissolved freshwater values.
5. Lowest EPA WQC for saltwater usually chronic total dissolved.
6. Typical dissolved saltwater values.
* Indicates total dissolved concentration combining both Cr (III) and Vr (VI) concentrations.

- Metallic or metal-containing sampling equipment, containers, labware (e.g. talc gloves containing high levels of zinc), reagents, and deionized water
- Improperly cleaned and stored equipment, labware, and reagents
- Atmospheric inputs such as dirt and dust from automobile exhaust, cigarette smoke, nearby roads, bridges, wires, poles, etc. (Even human contact can be a source of trace metals contamination. For example, it has been demonstrated that dental work (e.g., mercury amalgam fillings) in the mouths of laboratory personnel can contaminate samples that are directly exposed to exhalation.)

Clearly preventing ambient water samples from becoming contaminated during the sampling and analytical process is the greatest challenge faced in trace metals determinations. Over the last two decades, marine chemists have come to recognize that much of the historical data regarding the concentrations of dissolved trace metals in seawater are erroneously high because the concentrations reflect contamination from sampling and analysis rather than ambient levels. More recently, historical trace metals data collected from freshwater rivers and streams have been shown to be similarly biased due to contamination during sampling and analysis. Therefore, it is imperative that extreme care be taken to avoid contamination when collecting and analyzing ambient water samples for trace metals. If these clean chemistry procedures are not followed, the resulting data will most likely be considered invalid and not defensible.

The EPA has recently issued (April 1995) detailed guidance on sampling ambient water for trace metals at low WQC levels (Method 1669). A companion document provides guidance on evaluating the data quality of low level ambient water trace metal data. The U.S. Geological Survey (USGS) has issued its own guidance for clean sampling of ambient waters. Much of the guidance provided here has been adapted from these guidance documents.

13.3.2 Trace Organics. The approach for sampling trace organics in water samples is fundamentally the same as for trace metals but the level of effort required to obtain contamination-free samples is significantly less because most synthetic organic compounds being measured are not naturally occurring. Many trace organics may be absent or at extremely low concentrations in many parts of the environment and consequently, the chance for contamination is less. Also, the environmental concentrations of many organic compounds, which do occur naturally, are substantially lower than those for trace metals. An important exception is organics associated with the burning of fossil fuel or smoking, which are widespread human activities which must be controlled when sampling for trace organics.

The large majority of trace organic analyses of water involve determining the total concentration of the compound of interest which can be extracted from the sample using organic solvents (e.g. hexane, methylene chloride, etc.). The filtration of samples to determine dissolved organics is not commonly done since the total recoverable concentration is considered a better indicator of the amount of bioavailable compound present. Filtration is more often used to remove suspended solids which can interfere with sample analysis.

There is no consolidated guidance (such as method 1669 for trace metals) from the EPA for the clean sampling of trace organics. The procedures are scattered among the scientific literature and EPA methods and can vary depending on the trace organic compound to be determined. The guidance given here will be general. Prior to sampling for trace organics, the appropriate EPA analytical method(s) should be consulted for specific guidance on field sampling of the exact trace organics to be measured. The EPA is issuing new trace organics methods (1600 series) which will provide the latest guidance.

13.4 WHEN TO USE CLEAN SAMPLING PROCEDURES. Two of the most important factors in successfully implementing these procedures are:

- An awareness of potential sources of contamination
- Strict attention to work being performed

Therefore, it is imperative that the procedures described in this method be carried out by well-trained, experienced personnel. Documentation of training should be kept on file and be readily available for review.

13.4.1 Trace Metals. EPA method 1669 was written to cover all sampling situations including the collection of water samples for mercury (Hg) analysis at the sub-ppt range. As a result, this method includes many procedures which are unnecessary (i.e. overkill) for a project which does not require such low detection limits. This section provides practical guidance on when and how to tailor clean sampling procedures to meet the data quality objectives (DQO) of each monitoring study. Unnecessary procedures can be omitted to control costs without sacrificing data quality. Method 1669 as well as the 1600 series analytical methods are all performance based. The methods can be modified, including the use of less stringent procedures when appropriate, as long as all QA performance criteria are met. For example, equipment cleaning steps can be eliminated if equipment and field blanks remain below the target monitoring MDL.

Table 13-1 summarizes the lowest WQC for both freshwater (Column 4) and saltwater (Column 6) and the ML's (Column 3). The metals fall into two groups:

- **Mercury**, whose WQC (0.012 ppb) is so close to the natural background level that ultra-clean procedures are needed for all monitoring
- **All other metals**, whose WQC are often much higher than natural background levels requiring less stringent clean procedures for monitoring

A three tiered approach is recommended for implementing the procedures described in this chapter. The objective of this approach is to determine the optimum level of clean sampling effort required to meet the DQO's for the monitoring program of interest.

- Tier 1. Required MDL's for all metals to be measured are greater than 200 ppb. Use of existing technology-based effluent monitoring methods for the collection and analysis of water and wastewater should be adequate to produce accurate data.

- Tier 2. Required MDL of at least one metal to be measured is less than 200 ppb, but greater than Tier 3 target MDLs. Clean procedures must be used to produce accurate data for all metals of interest. This is the recommended minimum for clean sampling.

- Tier 3. Required MDL of at least one metal to be measured is less than Tiers 2 and 3 target MDLs. Ultra-clean procedures must be used to produce accurate data for all metals of interest. This is the suggested minimum for quantitating aqueous trace metals at ambient (background) levels.

The tiers are defined in terms of threshold MDL's which can vary among metals. **Table 13-2** summarizes threshold MDLs for the 13 priority pollutant metals. Tier 1 and Tier 2 MDLs are set at 200 ppb for all elements. This threshold represents an order of magnitude safety factor over the concentration (i.e. ~ 20 ppb) where sample contamination begins to be a significant concern for most metals. Tiers 2 and 3 threshold MDLs are based on field and laboratory experience and are set at the level for each element at which contamination (i.e. detectable blanks) begins to be observed using clean (less stringent) procedures. Unless two different sampling procedures are used, the metal with the lowest threshold MDL will determine the level of effort (tier) used to collect all samples.

Table 13-2 Threshold MDL's Defining the Level of Effort (Tier) Required to Collect Uncontaminated Aqueous Trace Metal Samples

	Threshold MDL Range (Fg/L = ppb)		
Element	Tier 1 Existing Methods	Tier 2 Clean	Tier 3 Ultra-Clean
Antimony	> 200.0	< 200.0 - 0.1	< 0.1
Arsenic (III)	> 200.0	< 200.0 - 0.1	< 0.1
Beryllium	> 200.0	< 200.0 - 0.05	< 0.05
Cadmium	> 200.0	< 200.0 - 0.05	< 0.05
Chromium (III)	> 200.0	< 200.0 - 0.1	< 0.1
Copper	> 200.0	< 200.0 - 0.1	< 0.1
Lead	> 200.0	< 200.0 - 0.07	< 0.07
Mercury	> 200.0	< 200.0 - 0.03	< 0.03
Nickel	> 200.0	< 200.0 - 0.1	< 0.1
Selenium	> 200.0	< 200.0 - 0.05	< 0.05
Silver	> 200.0	< 200.0 - 0.03	< 0.03
Thallium	> 200.0	< 200.0 - 0.1	< 0.1
Zinc	> 200.0	< 200.0 - 1.0	< 1.0

In the method sections of this document (i.e. **Sections 13.5 through 13.9**), two levels of guidance are offered. The first designated clean (Tier 2, recommended minimum) represents the level of effort that should be invested in the collection, processing, and analysis of any environmental sample where the required MDL is less than 200 ppb. The second level designated ultra-clean (Tier 3) represents an enhanced level of effort that may be needed if the Tier 2 level of effort fails to satisfy DQO. Tiers 2 and 3 threshold MDLs can be used as a guide to the concentrations where such enhanced effort may be needed.

13.4.2 Trace Organics. The distinction between clean and ultraclean procedures has less meaning for organics due to the reduced likelihood of contamination. The tiered level of effort approach is not used for trace organics in this guidance. Still, the level of effort for clean organics sampling is also tied to the target MDL needed for a given study. Greater effort and care are needed to collect a trace organics sample set with target MDLs in the low ppt range compared to a set with target MDLs in the ppb range.

13.5 SPECIALIZED FACILITIES AND EQUIPMENT.

13.5.1 Trace Metals.

13.5.1.1 Laboratory Space. The best way to control contamination is to completely avoid exposure of the sample and apparatus to contamination in the first place. Avoiding exposure means performing operations in an area known to be free from contamination. No high level trace metal samples are allowed in the portion of the lab reserved for clean trace metals work.

13.5.1.1.1 Tier 2 (Clean). As a minimum, a portion of the laboratory should be set aside for low level trace metals work and isolated from the rest of the area by some sort of vertical plastic barrier. An effort should also be made to control material falling from the ceiling into the isolated area. All metal fixtures should be replaced with plastic ones and all surfaces should be coated with epoxy paint to minimize airborne particulates. This area should include a fume hood for acid cleaning all labware. A class 100 laminar flow clean bench is desirable but not necessary as long as care is taken to avoid contamination whenever labware or samples are open to the laboratory atmosphere.

13.5.1.1.2 Tier 3 (Ultra-Clean). As a minimum, in addition to the Tier 2 requirements, a class 100 laminar flow clean bench is required and an entirely separate class 100 clean room is desirable. The EPA has recently issued guidance on constructing such cleanrooms in existing spaces.

13.5.1.2 Labware. The labware is the same for both Tier 2 and Tier 3 applications. The only difference between tiers is how the labware is cleaned. Also, the necessary labware should be set aside and used only for clean sampling work. All labware and other apparatus used for clean trace metals sampling must be non-metallic and free of material that may contain metals. Only the following materials should come in contact with samples: fluoropolymer, conventional or linear polyethylene, polycarbonate, polysulfone, polypropylene, or ultra-pure quartz. PTFE is less desirable than FEP because the sintered material in PTFE may contain contaminants and is susceptible to serious memory effects. Only fluoropolymer should be used for samples that will be analyzed for mercury because mercury vapors can diffuse in or out of other materials, resulting either in contamination or low-biased results. Glass and metal must not be used under any circumstance.

The following materials have been found to contain trace metals and must not be used to hold liquids that come in contact with the sample or must not contact the sample, unless these materials have been shown to be free of the metals of interest at the desired level: Pyrex, Kimax, methacrylate, polyvinylchloride, nylon, and Vycor. In addition, highly colored plastics, paper cap liners, pigments used to mark increments on plastics, and rubber all contain trace levels of metals and must be avoided.

Table 13-3 Reagent Water Quality for Clean Sampling

Quality Parameter	Type I	Type II	Type III
Bacteria, CFU/ml	10.0	1000.0	NA
pH	NA	NA	5.0-8.0
Resistivity, megohm-cm at 25EC	> 10.0	> 1.0	0.1
Conductivity, mho/cm at 25EC	< 0.1	1.0	10.0
SiO_2, mg/L	< 0.05	< 0.1	< 1.0
Total solids, mg/L	0.1	1.0	5.0
Total oxidizable organic carbon mg/L	< 0.05	< 0.2	< 1.0
Trace metals of interest (ng/L)	< 5.0	< 100.0	< 1000.0
Trace organics of interest (ng/L)	< MDL*	< ML*	< 500.0

NAIndicates not applicable

13.5.1.3 **Reagent Water. Table 13-3** defines three types of reagent water.

NOTE:

> The water types specified in **Table 13-3** are for clean sampling and testing as defined in this chapter. The water quality used must be specified as to reference and type in the Field Sampling Plan (e.g., ASTM Type I, SM Type I, or EPA Type I)

Large volumes of Type I and II reagent water are needed for labware cleaning and other activities associated with clean sampling for trace metals. Type I and II reagent water can be prepared by various methods including distillation, deionization, reverse osmosis, anodic/cathodic stripping voltammetry, or other techniques that remove the metal(s) and potential interferant(s).

* Indicates not detected at or above the value indicated for the analytical method being used to measure the trace organic(s) of interest. ML=3.18 times the MDL.

13.5.1.4 **Sampling Equipment.** The only distinction between Tier 2 and 3 sampling equipment is how the equipment is handled and cleaned. Depending on the sampling requirements no specialized sampling equipment may be needed. Manual grab sampling, if feasible, is an ideal sampling method since the sample is introduced directly into the sample container below the surface of the water. Other pole-mounted or submersible samplers (See **Section 13-8**) may be needed. All samplers should be completely metal-free and used only for clean sampling work. However, after manual grab sampling, the preferred method is to collect the samples by pumping with a peristaltic tubing pump because the sample is never exposed to the atmosphere. A battery powered tubing pump such as the Masterflex models 7533 or 7570 or equivalent is a good choice. If automated composite samplers are needed, an all plastic peristaltic tubing pump type sampler is required. At this time, only total recoverable (not dissolved) samples can be collected using this type of composite sampler.

13.5.2 Trace Organics.

13.5.2.1 **Laboratory Space.** A portion of the laboratory should be reserved for trace organics work and isolated from the rest of the laboratory by vertical barriers. Non-plastic work surfaces are preferred. The area must have a sink and fume hood for labware cleaning. An effort should also be made to control material falling from the ceiling into the isolated area.

No high level organics samples should be allowed in this area.

13.5.2.2 Labware. All labware and equipment should be glass (amber or clear as needed) or metal. Exceptions are bottle caps, stopcocks, etc., which should be lined with or constructed out of fluoropolymer. All labware should be reserved for trace organics work only.

13.5.2.3 Sampling Equipment. Manual grab sampling is the preferred method of field sampling. Other all metal grab samplers which can hold glass sample containers may be needed. If automatic composite samplers are needed, a system incorporating glass containers and glass or fluoropolymer tubing is required. All sampling equipment should be reserved for trace organics work.

13.6 HAZARDS AND SAFETY PRECAUTIONS.

Refer to **Chapter 3**, **Section 3.3.3** and **Appendix D** for specific hazard identification and appropriate reference for detailed control measures.

13.6.1 Trace Metals. No special hazards or safety precautions are associated with clean sampling of trace metals. Standard laboratory practices for safe handling of the strong mineral acids needed to clean the labware and apparatus must be observed. Personal safety during field sampling (e.g. life jackets, proper clothing, etc. (See **Chapter 3, Section 3.3.3** and **Appendix D** for specific hazard identification and appropriate reference for detailed control measures.)) on or near bodies of water is an important consideration.

13.6.2 Trace Organics. No special hazards or safety precautions are associated with clean sampling of trace organics. Standard laboratory practices for safe handling of volatile organic solvents must be observed. Personal safety during field sampling (e.g. life jackets, proper clothing, etc. (See **Chapter 3**)) on or near bodies of water is an important consideration.

13.7 PREPARATIONS FOR FIELD SAMPLING.

13.7.1 Trace Metals.

13.7.1.1 Reagent Water. Type III water should only be used for Tier 1 sampling and analysis which are not covered in this chapter. Type II water (i.e. generally called distilled-deionized) is used for all cleaning in both Tier 2 and Tier 3 work except for the final rinse of equipment. Type I water may be used for the final rinse of Tier 2 equipment and must be used for the final rinse of Tier 3 equipment. If cleaned sampling equipment is stored full with water or dilute acid, then Type I water must be used. Type I water is used for preparing standards and for all sample digestions and dilutions in Tier 2 and 3 work. Type I water is also used to prepare all equipment blanks. Also, a large carboy or other appropriate container filled with Type II water must be available in the field to collect field blanks.

It is worth noting, that even Type I water has measurable concentrations of metals. When doing Tier 3 work with MDL's in the low parts per trillion range (i.e. enhanced sensitivity techniques such as preconcentration), even the best Type I water may contribute a measurable laboratory method blank for some metals.

13.7.1.2 Reagent Chemicals. Four general grades of chemicals are commercially available for use in trace metals sampling and analysis:

- Technical grade (highest metal levels, not discussed here)
- Reagent grade
- Trace metal grade
- Ultrapure grade

The maximum contaminant levels of reagents (i.e. primarily mineral acids such as nitric and hydrochloric) are shown in **Table 13-4**. The use of the various grades of chemicals (i.e. mostly mineral acids) as a function of level of effort (tiers) is shown in **Table 13-5**. Reagent grade chemicals have been shown to contain concentrations of metals that will interfere with the determination of trace metals at ambient water quality criteria levels. As a result, trace metal grade and ultrapure grades are used almost exclusively. For Tier 3 level work, with highly sensitive analytical techniques (e.g. preconcentration), chemicals (other than acids) must undergo additional purification procedures to reduce the method blank to the lowest possible level.

Table 13-4 Maximum Containment Levels Allowed for Reagent Chemicals Used in Clean Sampling of Trace Metals

	Maximum Allowed Concentration (ppb)		
Containment	Reagent Grade	Trace Metal Grade	Ultrapure Grade
Iron	00.0	5.0	< 0.1
Sulfate	00.0	< 200.0	300.0
Potassium	00.0	< 20.0	< 0.2
Aluminum			< 0.01
Arsenic (and antimony as Arsenic)	.0	< 3.0	< 0.01
All other metals (including Al, Ag, Be, Cd, Cr, Cu, Hg, Ni, Pb, Sb, Se, Sn, Tl, V, Zn)	00.0		
Silver			
Beryllium		< 1.0	< 0.005
Cadmium		< 1.0	< 0.005
Chromium		< 1.0	< 0.005
Copper		1.0	< 0.005
Mercury		< 1.0	< 0.005
Nickel		< 0.5	< 0.1
Lead		0.4	< 0.005
Selenium		< 0.4	< 0.005
Tin			< 0.01
Thallium		< 1.0	< 0.005
Vanadium		< 20.0	< 0.005
Zinc		< 1.0	< 0.002
		< 1.0	< 0.02

Table 13-5 Grades of Reagent Chemicals to Use as a Function of Tier (Level of Effort) and Activity

Activity	Tier	Reagent Grade	Trace Metal Grade	Ultrapure Grade
Equipment cleaning	1	X		
	2-3		X	
Filter neutralization	1		X	
	2-3			X
Sample preservation	1		X	
	2-3			X
Sample digestions	1		X	
	2-3			X
Standards preparation	1		X	
	2-3			X
Sample dilutions	1		X	
	2-3			

13.7.1.3 <u>Labware Cleaning</u>. Regardless of construction, all materials that will directly or indirectly contact the sample must be cleaned using these procedures. All cleaning should be done in a designated cleaning area. For Tier 2 sampling, the cleaning can be done in an isolated part of the laboratory set aside for that purpose. For Tier 3 sampling, the minimum configuration is a clean bench in an isolated part of the laboratory. Powderless vinyl gloves, a lintless lab coat and hat should be worn at all times. Materials such as gloves, storage bags, and plastic wrap may be used new without additional cleaning unless the results of the equipment blanks pinpoint any of these materials as a source of contamination. In this case, either an alternate supplier or pre-cleaning is required. Serial numbers should be indelibly marked or etched on each piece of apparatus (including cartridge filters) so that contamination can be traced. Logbooks should be maintained to track the sample from the container, through the sampling process, to shipment to the laboratory. **Chain-of-Custody** (COC) procedures are also used to trace contamination to particular handling procedures or lab personnel. The procedure for cleaning bottles, labware, sampling equipment including filters, etc., is described below. Type II water and trace metal grade reagents should be used unless noted.

13.7.1.3.1 <u>Tier 2 (Clean, Recommended Minimum)</u>.

1. Wash inside and outside of equipment thoroughly in a 0.5% laboratory detergent solution. Rinse thoroughly with Type II water.
2. Fill to the rim with 1:2 hydrochloric acid (HCl) (trace metal grade,) and cap. Let soak at least 24 hours. Rinse at least three times with Type II water. A final rinse should be done with the ultrapure water (Type I) if available. Bottles can be stored empty. Acids should be discarded after 5-10 uses based on the level of metal accumulation.
3. For capsule filters, it is easiest to pump the acid and rinse solutions through the filter. The filter must be rinsed thoroughly to remove any entrained acid. The rinse water pH should be checked and the filter neutralized, if necessary, with ultrapure ammonium hydroxide.
4. C-flex or silicone tubing should be filled with 10% HCl and allowed to soak for 24 hours before being rinsed several times with water. Tubing

should be formed into loops and the ends mated using small pieces of c-flex tubing.

5. All equipment should be double bagged in clear polyethylene bags or polyethylene film for storage (in clean, closed coolers) and shipment to the field with a batch/lot identification tag. Storage time before use should be minimized.

13.7.1.3.2 Tier 3 (Ultra-Clean).

1. Same as Tier 2 except all cleaning should be done in a class 100 clean bench or clean room and labware should soak at least 96-168 hours or be heated to 50-60 EC for 24 hours.
2. Equipment (except filters and tubing) should be soaked 96-168 hours in 1:1 trace metal grade nitric acid or be heated to 50-60 EC for 24 hours between the detergent and HCl steps.
3. Equipment should be air-dried in the clean room.
4. Sample containers should be stored full of 0.1% (v/v) ultrapure HCl/Type I water.

13.7.1.4 Equipment Blanks. Prior to the use of any sampling equipment in the field, equipment blanks must be generated in the laboratory to demonstrate that the equipment is free from contamination. Two types of equipment blanks are required: bottle blanks and sampler check blanks. Equipment blanks must be run on each type of equipment that will be used in the field. If, for example, samples are to be collected using both a grab sampling device and the jar sampling device, then an equipment blank must be run on both pieces of equipment. Equipment blanks are generated in the laboratory by processing Type I water through the equipment using the same procedures that are used in the field (i.e. "clean hands/dirty hands" technique (See **Section 13-8**)). The equipment blanks must be analyzed using the appropriate analytical method. If any metal(s) of interest or any potentially interfering substance is detected in the equipment blanks at the minimum level required by the study, the source of contamination/interference must be identified and removed.

13.7.1.4.1 Tier 2 (Clean). One sample bottle from each cleaning batch should be filled with Type I reagent water and stored in the laboratory. If field blanks collected in bottles from the same batch show signs of contamination, then the bottle blank can be preserved and analyzed like a normal sample to help pinpoint the source of contamination. If the field blanks are free of contamination, there is no need to process the bottle blank further.

Sampler check blanks are generated in the laboratory by processing reagent water (Type I) through the equipment in the same manner as in the field. A sampler check blank should be performed on at least one representative piece of sampling equipment to be used in the field. The sampler check blank should be stored and analyzed, if necessary, as described for the bottle blank.

13.7.1.4.2 Tier 3 (Ultra-Clean). At least one sample bottle from each cleaning batch should be filled with Type I water and preserved as described in **Section 13-9**. The bottle should stand for at least 24 hours. Ideally, the bottles should stand for a time as close to the time the field samples will be stored in the bottles. After standing, the acidified water should be analyzed for signs of contamination. If any bottle shows signs of contamination, the problem must be identified, the cleaning procedures corrected or cleaning solutions changed, and all affected bottles recleaned. The bottles must be demonstrated to be free of the metals of interest before the equipment may be used in the field. Significant lead time between cleaning and use of equipment will be needed to comply with this requirement.

Sampler check blanks should be run on all equipment that will be used in the field. The equipment must be demonstrated to be free of the metals of interest before the equipment may be used in the field. If signs of contamination are found when the sampler check blanks are analyzed, each affected piece of equipment must be recleaned.

13.7.1.5. Sampling Plan. A written sampling plan should be prepared for every project involving clean metals prior to the performance of sampling. The plan need not be elaborate but should serve to focus attention on the DQO's required for the project. It is recognized that poorly planned sampling events compromise data quality. Therefore, a sampling plan is considered essential since it requires planning measures to address specific contaminant concerns. Furthermore, it outlines the goals of the sampling program in terms of acceptable blank concentrations. The elements of a satisfactory sampling plan include:

- Specific sampling methods to insure the necessary level of contamination control (clean or ultra-clean) is achieved
- The type of sample to be collected, the amount of sample needed, and the devices to be used (grab, surface, or subsurface samplers). Sufficient sample volume should be collected to allow for necessary quality control analyses, such as matrix spike/matrix spike duplicate analyses
- The number and type of equipment blanks, field blanks and other QA samples needed to meet project DQO's. The collection of equipment

blanks, field blanks, and field replicates is usually considered a minimum for clean sampling of trace metals. It is also desirable to include blind QC samples as part of the program.

- The acceptance/rejection criteria for the blank needed to meet project DQOs results as described in the QA/QC section.

In cases where clean metals data are collected for regulatory decision-making, compliance, or enforcement purposes, the sampling plan should be submitted to the regulating authority for review and written approval. Sampling should not be conducted until approval is received, and quality assurance objectives are agreed upon by both parties.

13.7.2 Trace Organics.

13.7.2.1. Reagent Water. Reagent water used must be free of the trace organics of interest at or above the MDL for the analytical method used. Consequently, the preparation of reagent water varies with the target analyte and MDL. However, Type I water quality is generally not a problem. Distilled water passed through an activated carbon filter is generally adequate for trace organics analysis.

13.7.2.2. Reagent Chemicals. Reagent grades of mineral acids and other chemicals (e.g. sodium sulfate) are adequate for trace organics analysis. Solvents should be pesticide grade and certified interference free. Solid phases (e.g. alumina), adsorbents (e.g. silica gel), and related chemicals should be chromatographic or high purity grade. Most analytical methods have specific procedures for cleaning these column related chemicals prior to analysis.

13.7.2.3 Labware Cleaning. Required cleaning procedures vary as a function of the target analyte and specific methods should be consulted. However, a general cleaning procedure for trace organics sampling includes:

1. Glassware should be rinsed with solvent and washed with a laboratory grade detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with fluoropolymer stopcocks, must be disassembled prior to detergent washing.
2. After detergent washing, glassware should be rinsed immediately, first with methanol and then with hot tap water. The tap water rinse is followed by another methanol rinse, then acetone and then methylene chloride.
3. Depending on the method, cleaned glassware should be combusted at 250-440 EC for 4 hours. However, some methods do not recommend combusting as a routine part of the cleaning procedure because repeated baking may cause the glassware to irreversibly adsorb some target analytes.

If filtration is required, a glass fiber filter (Gelman AE or equivalent) is recommended. The filter should be combusted prior to use.

13.7.2.4 Equipment Blanks. Equipment blanks are not required by most analytical methods. A laboratory method blank is used as a check for contamination.

13.7.2.5 Sampling Plan. The comments provided in **Section 13.7.1.5**, for trace metals, also apply here. The high level of blank samples required for trace metals (e.g. equipment and field) is felt to be unnecessary for the collection of contamination-free trace organics samples.

13.8 FIELD SAMPLING PROCEDURES.

Successful clean field sampling can be summarized in three points:

- Site and sampling platform selection to minimize the availability of external contaminants
- Use of clean equipment
- Minimize exposure of the sample to the external environment

13.8.1 Trace Metals. The descriptions below apply to both Tier 2 (clean) and Tier 3 (ultra-clean) levels of sampling efforts, except where Tier 3 only is indicated.

13.8.1.1 Site Selection. Selection of a representative site for surface water sampling is based on many factors including: study objectives, water use, point source discharges, nonpoint source discharges, tributaries, changes in stream characteristics, types of stream bed, stream depth, turbulence, and the presence of structures (bridges, dams, etc.). When collecting samples to determine ambient levels of trace metals, the presence of potential sources of metal contamination are of extreme importance in site selection.

Ideally, the selected sampling site will exhibit a high degree of cross-sectional homogeneity. It may be possible to use previously collected data to identify locations for samples that are well-mixed or are vertically or horizontally stratified. Since mixing is principally governed by turbulence and water velocity, the selection of a site immediately downstream from a riffle area will ensure good vertical mixing. Horizontal mixing occurs in constrictions in the channel. In the absence of turbulent areas, the selection of a site that is clear of immediate point sources, such as industrial effluents, is preferred for the collection of ambient water samples.

In order to minimize atmospheric trace metals contamination, ambient water samples should be collected from sites that are as far as possible (e.g., at least several hundred feet) from any metal supports, bridges, wires or poles. Similarly, samples should be collected as far as possible from regularly or heavily traveled roads. If it is not possible to avoid collection near roadways, it is advisable to study traffic patterns and plan sampling events during lowest traffic flow. Whenever possible, the sampling activity should occur as far as possible from these sources of airborne contamination. Areas where nearby soil is bare and subject to wind erosion should be avoided.

The sampling activity should be planned to collect samples known or suspected to contain the lowest concentrations of trace metals first, finishing with the samples known or suspected to contain the highest concentrations. For example, if samples are collected from a flowing river or stream near an industrial or municipal discharge, the upstream sample should be collected first, the downstream sample collected second, and the sample nearest the discharge collected last. If this sequence cannot be observed, contamination from carryover (high sample followed by low sample) might occur. When necessary, the sample collection system may be rinsed with dilute acid and Type II water between samples followed by collection of a field blank.

13.8.1.2 Field Contamination Control. The apparatus should be clean when received by the sampling team. If there are any indications that the apparatus is not clean (e.g., ripped storage bags), an assessment of the likelihood of contamination must be made. Sampling must not proceed if it is possible that the apparatus is contaminated. If the apparatus is contaminated, it must be returned to the laboratory or cleaning facility for proper cleaning before any sampling activity resumes. Having backup apparatus is recommended to avoid these delays.

The apparatus that will contact samples or blanks should only be opened or exposed to the atmosphere during the period of sampling. When not being used, the apparatus should be covered with clean plastic wrap, stored in the clean bench or in a plastic box or glove box, or bagged in clean, colorless zip-type bags. Apparatus that may not directly contact samples may still be a source of contamination. For example, clean tubing placed in a dirty plastic bag may pick up contamination from the bag and subsequently transfer the contamination to the sample. Therefore, it is imperative that every piece of the apparatus be properly stored when not in use.

Significant contamination of the apparatus may result when untreated effluents, in-process waters, landfill leachates, and other samples containing mid-to high-level concentrations of inorganic substances are processed. Clean sampling methods are not intended for application to these samples, and samples containing high concentrations of metals ($ 10 Fg/L) must not be collected, processed, or shipped at the same time as samples being collected for trace metals determinations.

Sampling personnel must wear clean, non-talc gloves during all operations involving handling of the apparatus, samples, and blanks. Only clean gloves may touch the apparatus. If another object or substance is touched, the glove(s) must be changed before again handling the apparatus. If it is even suspected that gloves have become contaminated, work must be halted, the contaminated gloves removed, and a new pair of clean gloves put on. Wearing multiple layers of clean gloves will allow the old pair to be quickly stripped with minimal disruption to the work activity. Shoulder-length gloves are needed if samples are to be collected by direct submersion of the sample bottle into the water or when sampling for mercury (Tier 3 only).

Only metal-free (e.g., fiberglass) boats should be used, along with wooden or fiberglass oars. A flat-bottom, Boston Whaler type boat is preferred because sampling materials can be stored with reduced chance of tipping over. Gasoline or diesel fueled boat motors should be avoided when possible because the exhaust can be a source of contamination. If the body of water is so large as to necessitate the use of a boat motor, the engine should be shut off at a distance far enough from the sampling point as to avoid contamination, and the sampling team should manually propel the boat to the sampling point. Samples should be collected upstream of boat movement. Before first use, the boat should be cleaned and stored in an area that minimizes exposure to dust and atmospheric particles. For example, cleaned boats should not be stored in an area that would allow exposure to automobile exhaust or industrial pollution. The boat should be frequently visually inspected for possible contamination. Immediately before use, the boat should be washed down with water from the sampling site away from any sampling points to remove any dust or dirt accumulation. After sampling, the boat should be returned to the laboratory or cleaning facility, cleaned as necessary, and stored away from any sources of contamination until next use.

Tier 3 sampling efforts require additional contamination control procedures. A field-portable glove bag (e.g., I2R, Model R-37-37H, non-talc or equivalent) should be used for all sample transfers. Alternately, a portable glove box may be constructed with a non-metallic (PVC pipe or other suitable material) frame and a frame cover made of an inexpensive, disposable, non-metallic material (e.g., a thin-walled polyethylene bag). An unlined, long-sleeved wind suit (e.g., Tyvek[7], Pamida, or equivalent) consisting of pants and jacket and constructed of nylon or other synthetic fiber should be worn when sampling for mercury to prevent mercury adsorbed onto cotton or other clothing materials from contaminating samples. The wind suit is washed by itself or with other wind suits only in a home or commercial washing machine and dried in a clothes drier. The clothes drier must be thoroughly vacuumed, including the lint filter, to remove all traces of lint prior to drying. After drying, the wind suit is folded and stored in a clean, polyethylene bag for shipment to the sample site.

13.8.1.3 Sample Collection Procedures. Sampling personnel should ideally approach the collection site from down current and downwind in order to prevent contamination of the sample by particles sloughing off the boat or equipment. If it is not possible to approach from both, the site should be approached from down current if sampling from a boat or approached from downwind if sampling on foot. When sampling from a boat, the bow of the boat should be oriented into the current (the boat will be pointed upstream). All sampling activity should occur from the bow.

If the samples are being collected from a boat, sampling personnel should create a stable workstation by arranging the cooler or shipping container as a work table on the upwind side of the boat. This work table and the upwind gunnel should be covered with plastic wrap or a plastic tablecloth. If necessary, duct tape should be used to hold the wrap or cloth in place.

All operations involving contact with the sample bottle and with transfer of the sample from the sample collection device to the sample bottle (if the sample is not directly collected in the bottle) are handled by the individual designated as "clean hands". "Dirty hands" is responsible for all activities that do not involve direct contact with the sample. Initially, this appears to be a fairly clear-cut and separate division of responsibilities. In fact, the completion of the entire protocol may require a good deal of coordination and practice (e.g., "dirty hands" must open the box or cooler containing the sample bottle and unzip the outer bag; "clean hands" must reach into the outer bag, open the inner bag, remove the bottle, collect the sample, replace the bottle lid, put the bottle back into the inner bag, and zip the inner bag. "Dirty hands" must close the outer bag and place it in a cooler). To minimize unnecessary confusion, a third person should be available to complete the necessary sample documentation (e.g., to document sampling location, time, sample number, etc). Otherwise, the sample documentation activity must be performed by "dirty hands".

Extreme care must be taken during all sampling operations to minimize exposure of the sample to human, atmospheric, and other sources of contamination. Care must be taken to avoid breathing directly on the sample. Whenever possible, the sample bottle should be opened, filled, and closed while submerged.

If the sample is to be analyzed for dissolved metals, it should be filtered as described in **Sections 13.8.1.7 and 13.8.1.8**. After each sample is collected, the sample number is documented in the sampling log, and any unusual observations concerning the sample and the sampling are documented.

13.8.1.4 Manual Grab Sampling. Collecting samples directly into the sample container is the simplest procedure. This procedure has the least potential for contamination because it requires the least amount of equipment and handling.

1. At the site, all sampling personnel must put on clean gloves prior to commencing sample collection activity, with "clean hands" donning shoulder-length gloves. If samples are to be analyzed for mercury, sampling personnel must also put their pre-cleaned windsuits on at this time (Tier 3 only). Note that "clean hands" should put on the shoulder-length polyethylene gloves and both "clean hands" and "dirty hands" should put on the PVC gloves. "
2. Dirty hands" must open the cooler or storage container, remove the double bagged sample bottle from storage, and unzip the outer bag. Next, "clean hands" opens the inside bag containing the sample bottle, removes the bottle, and reseals the inside bag. "Dirty hands" then reseals the outer bag.

3. “Clean hands” unscrews the cap and, while holding the cap upside down, discards the dilute acid solution from the bottle into a carboy for wastes. “Clean hands” screws the cap on the

bottle, shakes the bottle several times, and empties the rinsate away from the site. After two more rinsings, “clean hands” holds the bottle under water and allows it to fill with sample. After the bottle has filled (i.e., when no more bubbles appear), and while the bottle is still inverted so that the mouth of the bottle is still underwater, “clean hands” replaces the cap of the bottle. In this way, the sample has never contacted the air.

4. Once the bottle lid has been replaced, "dirty hands" re-opens the outer plastic bag, and "clean hands" opens the inside bag, places the bottle inside it, and zips the inner bag. "Dirty hands" zips the outer bag.

13.8.1.5 <u>Grab Sampling Device</u>. The following steps detail sample collection using the grab sampling device shown in **Figure 13-2**. The procedure is indicative of the "clean hands"/"dirty hands" technique that must be used with alternative grab sampling devices such as that shown in **Figure 13-3**.

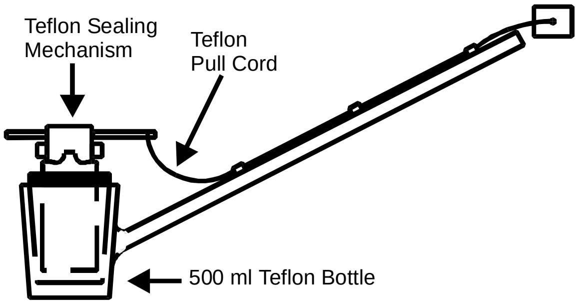

Figure 13-2 Grab Sampling Device

1. Sampling personnel put on gloves (and windsuits, if applicable) and handle bottles as with manual collection (See **Section 13.8.1.4**).
2. "Dirty hands" removes the sampling device from its storage container and opens the outer polyethylene bag. "Clean hands" opens the inside polyethylene bag and removes the sampling device. Ideally, a sample bottle will have been pre-attached to the sampling device in the class 100 clean room at the laboratory. If it is necessary to attach a bottle to the device in the field "clean hands" performs this operation inside the field-portable glove bag (Tier 3 only).
3. "Clean hands" changes gloves. "Dirty hands" submerges the sampling device to the desired depth and pulls the fluoropolymer pull-cord to bring the seal plate into the middle position so that water can enter the bottle. When the bottle is full (i.e., when no more bubbles appear), "dirty hands" pulls the fluoropolymer cord to the final stop position to seal off the sample and removes the sampling device from the water.

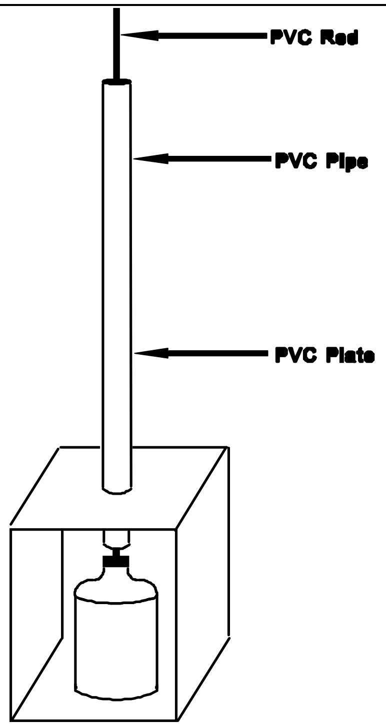

Figure 13-3 Grab Sampling Device

4. "Dirty hands" returns the sampling device to its large inner plastic bag, "clean hands" pulls the bottle out of the collar, unscrews the bottle from the sealing device, and caps the bottle. "Clean hands" and "dirty hands" then return the bottle to its double-bagged storage as described in **Section 13.8.1.4**.
5. "Clean hands" removes the closing mechanism from the body of the grab sampler, rinses the device with reagent water , places it inside a new clean plastic bag, zips the bag, and places the bag inside an outer bag held by "dirty hands". "Dirty hands" zips the outer bag and places the double bagged closing mechanism in the equipment storage box.
6. "Clean hands" seals the large inside bag containing the collar, pole, and cord and places the bag into a large outer bag held by "dirty hands". "Dirty hands" seals the outside bag and places the double bagged sampling device into the equipment storage box.

13.8.1.6 <u>Depth Sampling Device</u>. The following steps detail depth sampling using a jar sampling device (See **Figure 13-4**).

1. Sampling personnel put on gloves (and windsuits, if applicable) and handle bottles as with manual collection (See **Section 13.8.1.4**).

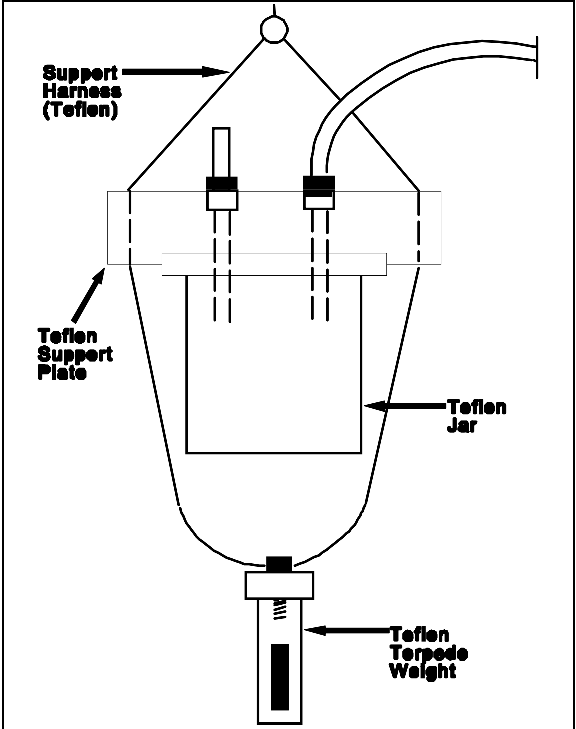

Figure 13-4 Jar Sampling Device

2. “Dirty hands" removes the jar sampling device from its storage container and opens the outer polyethylene bag. "Clean hands" opens the inside polyethylene bag and removes the jar sampling apparatus. Ideally, the sampling device will have been pre-assembled in a class 100 clean room at the laboratory. If, however, it is necessary to assemble the device in the field, then "clean hands" must perform this operation inside a field-portable glove bag (Tier 3 only).
3. While "dirty hands" is holding the jar sampling apparatus, "clean hands" connects the pump to the 1/4-inch OD flush line. Dirty hands" lowers the weighted sampler to the desired depth. "Dirty hands" turns on the pump allowing a large volume (>2 liters) of water to pass through the system.
4. After stopping the pump, "dirty hands" pulls up the line, tubing, and device and places them into either a field-portable glove bag or a large, clean plastic bag as they emerge. Both "clean hands" and "dirty hands" change gloves. Sampling personnel remove a sample bottle from storage, and "clean hands" places the bottle into the glove bag. "Clean hands" tips the sampling jar and dispenses the sample through the short length of fluoropolymer tubing into the sample bottle. Once the bottle is filled, "clean hands" replaces the cap of the bottle, returns the bottle to the inside polyethylene bag, and zips the bag. "Clean hands" returns the zipped bag to the outside polyethylene bag held by "dirty hands". "Dirty hands" zips the outside bag.

13.8.1.7 **<u>Continuous Pump Sampling</u>.** Next to manual grab sampling, continuous-flow sampling, using a submersible or peristaltic pump, is the sampling method least susceptible to contamination. The continuous-flow sampling system uses a peristaltic pump to pump sample to the boat or to shore through the SEBS-resin or PTFE tubing. The same set of precleaned pump tubing may be used to collect samples at more than one site if one is able to sample from low concentration to high concentration sites (See **Section 13.8.1.1**). A new set of precleaned pump tubing is needed for each different site sampled (Tier 3 only).

1. Prior to putting on wind suits or gloves, sampling personnel remove the bags containing the pump, SEBS-resin or FEP tubing, batteries, plastic wrap, wind suits, and, if samples are to be filtered, the filtration apparatus from the coolers or storage containers in which they are packed.
2. "Clean hands" and "dirty hands" put on the wind suits (Tier 3 only) and PVC gloves. "Dirty hands" removes the pump from its storage bag, and opens the bag containing the SEBS-resin or FEP tubing. "Clean hands" installs the tubing while "dirty hands" holds the pump. "Clean hands" immerses the inlet end of the tubing in the sample stream.
3. Both "clean hands" and "dirty hands" change gloves. "Clean hands" also puts on shoulder length polyethylene gloves. "Dirty hands" turns the pump on and allows the pump to run for 5 - 10 minutes or longer to purge the pump and tubing.
4. If the sample is to be filtered, "clean hands" installs the filter at the end of the tubing, and "dirty hands" sets up the filter holder on the gunwhale. The filtration apparatus should not be attached until immediately prior to sampling to prevent buildup of particulates from clogging the filter.
5. The sample is collected by rinsing the sample bottle and cap three times and collecting the sample from the flowing stream.

13.8.1.8 **<u>Sample Filtration</u>.** Filtration for dissolved trace metals must be done in the field as soon after the sample is collected as possible. Cartridge-type 0.45 :m tortuous path filters are recommended (Gelman 12175 Supor or equivalent). Filters cannot be re-used because of the risk of contamination from trapped particles and the risk of changes in the filtration characteristics due to particle loading. Also, the difficulty and risk associated with failing to properly clean these devices far outweighs the cost of purchasing new equipment. The filtration procedure described below is used for samples collected using the manual (See **Section 13.8.1.4**), grab (See **Section 13.8.1.5**), or jar (See **Section 13.8.1.6**) collection systems. In-line filtration using the continuous-flow approach has already been described in **Section 13.8.1.7**. Because of the risk of contamination, it is recommended that samples for mercury be shipped unfiltered via overnight courier and filtered upon receipt at the laboratory (Tier 3 only).

1. Set up the filtration system inside the glove bag (Tier 3 only), using the shortest piece of pump tubing as is practical. Place the peristaltic pump immediately outside of the glove bag and poke a small hole in the glove bag for passage of the tubing. Also, attach a short length of tubing to the outlet of the capsule filter.
2. "Clean hands" removes the water sample from the inner storage bag using the technique described in **Section 13.8.1.4** and places the sample inside the glove bag. "Clean hands" also places two clean empty sample bottles, a bottle containing reagent water, and a bottle for waste in the glove bag.
3. "Clean hands" removes the lid of the reagent water bottle and places the end of the pump tubing in the bottle. "Dirty hands" starts the pump and passes approximately 200 mL of Type II water through the tubing and filter into the waste bottle. "Clean hands" then moves the outlet tubing to a clean bottle and collects the remaining reagent water as a blank. "Dirty hands" stops the pump.
4. Clean hands" removes the lid of the sample bottle and places the intake end of the tubing in the bottle. "Dirty hands" starts the pump and passes approximately 50 mL through the tubing and filter into the remaining clean sample bottle and then stops the pump. "Clean hands" uses the filtrate to rinse the bottle, discards the waste sample, and returns the outlet tube to the sample bottle.
5. "Dirty hands" starts the pump and the remaining sample is processed through the filter and collected in the sample bottle. "Clean hands" replaces the lid on the bottle, returns the bottle to the inside bag, and zips the bag. "Clean hands" then places the zipped bag into the outer bag held by "dirty hands."
6. "Dirty hands" zips the outer bag, and places the double bagged sample bottle into a clean, ice-filled cooler for immediate shipment to the laboratory.

13.8.1.9 Field QA Samples.

13.8.1.9.1 Field Blanks. Field blanks are the most important single QC sample associated with clean sampling of trace metals. They are used to demonstrate that sample contamination has not occurred during field sampling and processing. At least one (1) total recoverable (no filter) field blank must be generated for every ten (10) samples that are collected at a given site. Field blanks are collected prior to sample collection. A separate (second) field blank should be collected using a filter cartridge if dissolved samples are also being collected.

Field blanks are generated by filling a large carboy or other appropriate container with Type II water in the laboratory, transporting the filled container to the sampling site, processing the water through each of the sample processing steps and equipment (e.g., tubing, sampling devices, filters, etc.) that will be used in the field, collecting the field blank in one of the sample bottles, and handling the bottle in the same manner as a normal, unknown field sample.

13.8.1.9.2 Field Duplicates. In order to assess the precision of the field sampling and analytical processes, at least one (1) total recoverable field duplicate sample must be collected for every ten (10) samples that are collected at a given site. A separate (second) field duplicate should be collected if dissolved (filtered) samples are also being collected. The field duplicate is collected either by splitting a larger volume into two aliquots in the glove box, by using a sampler with duel inlets that allows simultaneous collection of two samples, or by collecting two samples in rapid succession.

13.8.2 Trace Organics.

13.8.2.1 Site Selection. The considerations discussed in **Section 13.8.1.1** also apply to the clean sampling of trace organics. Probably the most important consideration is to collect trace organics samples as far as possible from regularly or heavily traveled roads or parking lots.

13.8.2.2 Field Contamination Control. Although the possibility of contamination is less for trace organics, many of the considerations discussed in **Section 13.8.1.2** also apply here. All sampling equipment should be clean and properly stored until used. If there is any doubt about the cleanliness of any piece of sampling equipment it should not be used. Exposure of sampling equipment to the atmosphere and surrounding environment should be minimized. Considering wind direction when sampling is especially important when sampling from a motorized boat. Exhaust fumes represent a significant source of potential contamination.

13.8.2.3 Sample Collection Procedures. The "clean hands"/"dirty hands" approach is not required for clean sampling of trace organics. However, a well coordinated sampling effort will minimize mistakes. Also, vinyl gloves should not be worn. Manual grab sampling is the preferred method of sampling for surface waters. The manual grab method described for trace metals (See **Section 13.8.1.4**) can be used to collect trace organics except that the sample bottle is not pre-rinsed with the water to be sampled prior to collecting the final sample. Sample documentation requirements are obviously the same for trace organics sampling. Aqueous trace organics samples should be kept in the dark and cooled to 0-4 EC as soon as possible after collection. If possible, they should be stored on ice immediately after collection until they can be refrigerated.

13.8.2.4 Continuous Pump Sampling. This approach should be used to collect trace organics samples at depth. Only fluoropolymer or glass tubing should be used. Except for the two-hands approach and pre-rinsing the sample bottle, the trace metals procedure (See **Section 13.8.1.7**) can be used here.

13.8.2.5 Sample Filtration. Filtration of trace organics is not commonly done and should not be done in the field. Cartridge type filters used for trace metal samples cannot be used for trace organics samples. If filtration is necessary, it should be done after the samples are returned to the laboratory and combusted glass fiber filters as described in **Section 13.7.2.3** should be used.

13.8.2.6 Field QA Samples. No field QA sample is specifically required by trace organic analytical methods. However, for low detection level work (i.e. less than 1 ppb), it is recommended that at least one field (trip) blank be collected. An aliquot of reagent water is placed in a precleaned, glass sample container in the laboratory (trip blank), or in the field (field blank), and treated as a sample in all respects, including contact with the sampling devices and exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

13.9 POST-SAMPLING PROCEDURES. These procedures (i.e. preservation and storage) stabilize the field samples until they can be analyzed.

13.9.1 Trace Metals. Field preservation is not necessary for dissolved metals, except for trivalent and hexavalent chromium, provided that the sample is preserved in the laboratory and allowed to stand for at least two days in order to allow the metals adsorbed to the container walls to redissolve. Field preservation is advised for hexavalent chromium in order to provide sample stability for up to 30 days. Mercury samples should be shipped via overnight courier and preserved upon receipt at the laboratory.

If field preservation is required, preservation must be performed in the glove bag or in a designated clean area, with gloved hands, as rapidly as possible to preclude particulates from contaminating the sample. For preservation of trivalent chromium, the glove bag or designated clean area must be large enough to accommodate the vacuum filtration apparatus, and an area should be available for setting up the wrist-action shaker. It is also advisable to set up a work area that contains a "clean" cooler for storage of clean equipment, a "dirty" cooler for storage of "dirty" equipment, and a third cooler to store samples for shipment to the laboratory. Preservation of aliquots for metals other than trivalent and hexavalent chromium: Using a disposable, pre-cleaned, plastic pipette, add 5 mL of a 10 percent solution of ultra-pure nitric acid reagent water per liter of sample. This will be sufficient to preserve a neutral sample to pH <2. Preservation can be done with care in the open laboratory (Tier 2) or in a clean bench or clean room (Tier 3). Trace metal samples should be stored in double plastic bags until processed for analysis.

13.9.2 Trace Organics. Aqueous trace organics samples should be maintained at 0-4 EC in the dark from the time of collection until extraction. Preservation of the sample varies with the target analyte and the analytical method to be used should be consulted. Preservation can involve adding acids or solvents such as methylene chloride.

13.10 SAMPLING EQUIPMENT LIST. Chapter 4, Section 4.8 provides a generic sampling equipment list applicable to most sampling events. The following list provides additional specific equipment applicable to Clean Sampling.

Safety Equipment, as required

Tier 2 Clean Sampling equipment and Lab facilities and equipment

Tier 3 Clean Sampling equipment and Lab facilities and equipment

Reagent Water, Type I, II or III as defined by the FSP

Chemical Reagents of the Grade and Quality defined in the SAP

Equipment cleaning and handling procedures consistent with SAP

Contamination control equipment and procedures consistent with SAP

Grab Sampling Device

Depth Sampling Device

Jar Sampling Device

Submersible or peristaltic pump for Continuous Sampling

Sample Filtration equipment

Cartridge-type 0.45 Fm tortuous path filters

Field Blanks

Equipment Blanks

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 14

FIELD TESTING

14.1 PURPOSE. This chapter provides guidance for performing testing in the field.

14.2 SCOPE. The scope of this chapter includes a discussion of field screening and field testing and gives examples of Standard Operating Procedures (SOPs) for several analyses that are required by various regulations to be performed in the field. Other analyses may be performed in the field, but are not required by regulations to be done in the field.

14.3 BACKGROUND. Analysis in the field is required for some parameters because of possible sample deterioration during transport to a laboratory, leading to potential erroneous results. Among the parameters requiring field testing are pH, Temperature, Residual Chlorine, Specific Conductance, Oxidizer Test, and Dissolved Oxygen. These tests may be performed as stand alone tests or in conjunction with additional sampling requirements.

14.4 TRAINING. Personnel who will be performing any field testing must have been trained and demonstrated proficiency in the Standard Operating Procedure for that test. Training and proficiency must be documented following the Uniform Standards for Sampling, OPNAVINST 5090.1B, *Environmental and Natural Resources Program Manual,* Chapter 25-4.2.

14.5 FIELD TESTING VERSUS FIELD SCREENING. The measurement of samples in the field may be performed by field screening techniques or field testing methods. The data quality objectives should dictate the type of measurement system.

14.5.1 Field Testing. Field testing is performed using the same analytical methods as a fixed laboratory. Data generated for compliance must meet the same rigorous quality control standards as a fixed laboratory. In addition, the environmental and site conditions must be recorded to ensure that future data comparability is based on the same test conditions. These environmental conditions are more constant in a fixed laboratory but they are extremely variable in the field. Recording parameters such as temperature, humidity, barometric pressure and test site location is critical when using the data for comparison or when repeating the test procedure. Examples of Field testing Standard Operating Procedures (SOPs) are in **Paragraph 14.7**.

14.5.2 Field Screening. Field screening procedures are often qualitative or semi-quantitative techniques that can be utilized in the field to give a quick determination of the presence, magnitude or absence of a pollutant. This minimizes the expense and extended turnaround time required to analyze samples in a laboratory. Since the Quality Control and analytical sophistication in field screening is not controlled to the same extent as laboratory testing, a cross section of additional samples may need to be submitted to a laboratory for confirmatory quantitation.

There are 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 demonstrated to ensure approval of the screening procedures for use by the regulatory authority. Positive and negative measurements are usually acceptable as long as the negative is below the action levels 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.

14.6 FIELD OPERATIONS. Field operations that include field screening or field testing should consider the following:

- Quality assurance manual
- Documented quality control procedures
- Test methods
- Calibration procedures
- Reference standards
- Materials and materials preparation
- Sampling storage
- Sample handling
- Sample traceability
- Quality control samples
- Control charts
- Data quality criteria
- Detection limits
- Record keeping
- Data reporting procedures
- Environmental conditions

- Facilities conditions and control
- Personnel qualifications
- Disposal
- Safety

14.7 SAMPLE OPERATING PROCEDURES. The remainder of this chapter provides sample Standard Operating Procedures for:

- Specific Conductance (Field Testing)
- Field Determination of Total Residual Chlorine
- Field pH Measurements Using pH Paper
- Field pH of Aqueous Samples by Electrometric Measurement
- Oxidizer Field Testing for Cyanide Samples
- Field Temperature Determination
- Calibration & Maintenance of a Photvac Photo Ionization Detector

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NAVY 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 to this manual 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.

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 waters, domestic and industrial wastes, and acid rain.. This procedure is for field testing where needed, however some regulations do allow this test to be performed in the laboratory. The Resource Conservation Recovery Act (RCRA) and the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) require analyses to be performed within 24 hours. Drinking water and waste water programs allow a 28 day holding time until analysis. See these regulations for sample preservation.

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. The ability of the solution is related to the ions present in the aqueous solution. Ground water sampling wells must have been previously installed.

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^o C. If samples are analyzed at different temperatures, corrections must be made and results reported at 25^o C.

4.0 INTERFERENCES

4.1 Platinum electrodes can degrade and cause erratic results. When this happens, as evidenced by erratic results or flaking off 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 to 1000 μmhos per cm) with Conductivity Cell (cell constant of 1.0, YSI #3403 or equivalent). Current METCAL calibration 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 shall be checked annually against a NIST traceable thermometer. Dial type thermometers shall be checked at least quarterly against a NIST traceable thermometer.)

5.4 Field log book/field notes.

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 0.7456gm 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 1413 µmhos per cm at 25° C. This solution can be prepared by laboratory personnel or equivalent NIST traceable reference standard may also be purchased.

6.2 Distilled water. If doing RCRA Method 9050, deionized distilled water is required with conductivity of less than 1 uhmo per cm.

7.0 HAZARDS and SAFETY PRECAUTIONS:

7.1 Refer to the Material Safety Data Sheet (MSDS) for details on chemical hazards and personal protection.

7.2 Minimum personal protective equipment (PPE), (i.e. chemical safety goggles and appropriately selected protective gloves), should be worn. Other PPE will be dictated by the specific hazards of the sampling site (i.e. safety shoes, hard hat, respirator).

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 Insure conductivity cell is correctly attached to conductivity meter.

8.3 Follow the directions of the manufacture for the operation of the instrument.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Prior to sampling (Note: This procedure to be performed only by properly trained sampling personnel).

9.1.1 Verify conductivity meter has current METCAL calibration

9.1.2 Calibrate conductivity meter prior to use, using KCl solution (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	*Micromhos/cm*
21	*1305*
22	*1332*
23	*1359*
24	*1386*
25	*1413*
26	*1441*
27	*1468*
28	*1496*

9.1.3 Enter 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 manufacturers' 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.

10.0 PROCEDURE

10.1 Obtain sample according to 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^{o} C, make temperature correction in accordance with the following guidelines:

- *If temperature of the sample is below 25^{o} C, add 2% of the reading per degree.*
- *If temperature is above 25^{o} C, subtract 2% of the reading per degree.*

The more the sample temperature of the measurement deviates from 25^{o} C., the greater the uncertainty in applying the temperature correction.

10.8 Record results in Field Log Book/Field Notes.

- *Sample ID*
- *Sample temperature ^{o}C*
- *Meter reading, umhos/cm*
- *Corrected meter reading, umhos/cm at 25^{o} C*
- *Signature and date*
- *SOP Reference name/number*

10.9 Recheck calibration, with the KCL solution (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 (paragraph 6.1) after approximately every 15 samples per RCRA method #9050 (paragraph 8.2).

11.5 Run 1 duplicate sample for every 10 samples per RCRA method #9050 (paragraph 8.2).

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^o C) Editorial Rev., 1982. Applicable to drinking water and wastewater.

12.2 Test Methods for Evaluating Solid Wastes. SW-846, September, 1986. Method 9050 Specific Conductance. Applicable to groundwater wells for RCRA monitoring.

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NAVY ENVIRONMENTAL SAMPLING AND FIELD TESTING PROCEDURE

STANDARD OPERATING PROCEDURE

FIELD DETERMINATION OF TOTAL RESIDUAL CHLORINE

PREP BY:
APPROVED BY:
EFFECTIVE DATE:

CAUTION

This Standard Operating Procedure has been prepared as an example to this manual 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.

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 with 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 waste water samples. Residual chlorine in drinking water must meet specified requirements. In waste water 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 is 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 colormetric 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 Stop Watch or Wrist Watch.

5.7 Various Appropriate Pipettes.

5.8 100 mL Volumetric Flasks.

5.9 Field Log Book / Field Notes

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 g KMNO4 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 six months. This reagent will be prepared by laboratory personnel or purchased from commercial sources.

6.3 DPD Total Chlorine Reagent Powder Pillows. (Purchased from HACH)

6.4 Sodium Hydroxide, 0.1N. This reagent will be prepared by laboratory personnel or purchased from commercial sources.

6.5 Nitric Acid, 0.1N. This reagent will 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 Material Safety Data Sheet (MSDS) for details on chemical hazards and personal protection.

7.2 Minimum personnel protective equipment (PPE) will be 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 (para 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 a 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 a 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) to (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.,3.,4.,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

8.3.1 Once the meter has been checked to ensure that it is in an 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 three points that bracket the 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 made in Field Log Book/Field Notes 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.

Note: Prior to beginning this procedure, ensure reagents are not beyond expiration date.

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 twenty seconds. Allow at least three minutes but not more than six 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 then 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 Field Log Book/Field Notes or Data Sheet 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 Field Log Book/Field Notes. Refer to the calibration table to determine the true values associated with the meter readings and record in Field Log Book/Field Notes 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 and 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 adjust the pH to within this range.

9.1.3 Calibration curves shall be established for each meter using the procedure of paragraph 8.2 prior to initial use and updated annually.

9.1.4 Prior to daily use, standardize meter using standardization procedure of 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 Field Log book or 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 may also be retained to determine if an interference exists, or for reanalysis using a different test method.

10.0QUALITY ASSURANCE FOR HACH DR100

10.1Ten percent of the samples will be analyzed in duplicate (split samples). Ten percent of the samples will have duplicate samples taken and analyzed (field duplicates). Record data in Field Log Book/Field Notes or appropriate Data Sheet. Follow QA/QC manual for reporting results.

10.2A 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 meter reading 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.

10.3The 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 instruments when the "Low Battery" indicator is lit. If this indicator light remains lit after batteries have been replaced, then this 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 cellholder 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 sample cell holder and sample cell should be kept as clean and free of scratches as possible. After each test, the sample cells used 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, 19th Edition; Section 4500-C1.G, DPD Colorimetric Method.

11.2 DR100 HACH Colorimeter Total Chlorine Test Kit Instruction Manual.

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	*(mg/L)*	*Meter (mg/L)*	*Corrected*	*Initials/Date/Time*

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NAVY 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 to this manual 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.

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 a corrosive or non-corrosive (pH less than 2 or greater than 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 or not 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 dropwise to the first sample until a pH change is observed by noting the color change.

4.2.4 Add base dropwise 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 Field Log Book/Field Notes.

5.0 EQUIPMENT:

5.1 __PPE (see para 7)
__A sample collection device, if necessary
__Sample containers, if necessary
__Field Log Book/Field Notes

6.0 REAGENTS AND MATERIALS

6.1 __Wide range pH paper
__Narrow range pH paper (every 0.5 pH unit)
__Certified pH buffers
__Dilute acid (e.g., 1:4 HCl) for identification of interferences
__Dilute base (e.g., 0.1N NaOH) for identification of interferences
__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., ground water, surface water, etc.), minimum PPE is safety glasses.

7.5 Consult Material Safety Data Sheet (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 which has been calibrated with certified buffers.

8.2 Document verification in the Field Log Book/Field Notes.

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 Field Log Book/Field Notes.

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 Field Log Book/Field Notes.

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 Field Log Book/Field Notes.

9.2.6 pH measurements can be used to define a waste as a corrosive or non-corrosive only if the measured values differ 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.

10.0QUALITY CONTROL

10.1All pH determinations must be performed in duplicate.

10.2Each batch of pH paper must be verified versus certified pH buffers or a pH meter which has been calibrated with certified pH buffers.

11.0REFERENCE

11.1SW-846 Test Method for Evaluating Solid Wastes, 3rd Edition, Update IIA, September 1994. Method 9041.

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NAVY 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 to this manual 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.

1.0 TITLE: SOP 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 effects both the electrode output and the sample itself. Therefore instrument calibration and sample analysis should be performed as near to 25^o 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 Field Log/Data Sheets.

6.0 REAGENTS AND MATERIALS

6.1 pH 4.0, 7.0, 10.0 @ 25^o C buffer solutions (buffer solutions to bracket expected sample values).

6.2 pH electrode filling solution (follow manufacturer instructions).

6.3 pH electrode storage solution (follow manufacturer 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 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 6.

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 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 two 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.

8.10The electrode slope (in percent) is then displayed in the main field with SLP in the lower field. The meter automatically advances to MEASURE mode.

8.11Record the slope in the Field Log Book/Field Notes. The slope shall be from 95% to 105%. If the slope is less than 95% or more than 105%, then recalibrate the pH meter.

8.12Rinse 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.

8.13Rinse 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.

8.14Record calibration date and pH meter identification number in the appropriate Field Log Book/Field Notes.
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 is displayed. Record the pH, sample identification, and sample time in the Field Log Book/Field Notes.

9.6 If elemental analytes are to be determined, then preserve the remainder of the sample by acidifying with nitric acid to a pH of _ 2.

10.0QUALITY CONTROL:

10.1The electrode shall be rinsed between samples.

10.2Buffers shall be NIST traceable and shall have a current shelf life.

10.3pH meters shall have a current METCAL calibration sticker.

10.4Calibration of mercury thermometers shall be checked at least annually against a NIST (NBS) - traceable certified thermometer.

10.5The calibration of dial - type thermometers shall be checked at least quarterly against a NIST (NBS) - traceable thermometer.

11.0REFERENCES:

11.1Standard Methods for the Examination of Water and Wastewater, 19th Edition: Section 1060, Collection and Preservation of Samples and Section 450-0-H, pH Value.

11.2Orion Research pH Meter Model 230A Instruction Manual.

11.3U.S. Environmental Protection Update No. 1, Test Methods for Evaluating Solid Waste, Section 9, Miscellaneous Methods, pH Measurement (5.5), Method 9040.

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 (to 25^{o} C)*	*pH CORRECTED*
7.0	____________	______________	______________
4.0	____________	______________	______________
10.0	____________	______________	______________

SLOPE _________

ANALYST SIGNATURE AND DATE: _______________________________

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NAVY ENVIRONMENTAL SAMPLING AND FIELD TESTING PROCEDURE

STANDARD OPERATING PROCEDURE

OXIDIZER FIELD TESTING FOR CYANIDE SAMPLES

PREP BY:
APPROVED BY:
EFFECTIVE DATE:

CAUTION

This Standard Operating Procedure has been prepared as an example to this manual 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.

1.0 TITLE: Standard Operating Procedure for Oxidizer Field Testing for Cyanide Samples.

2.0 SCOPE AND APPLICATION:

2.1 This Standard Operating Procedure 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 presents 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
__Sample containers
__Field Log Book/Field Notes

6.0 REAGENTS AND MATERIALS

6.1 __Potassium iodide - starch test paper (KI starch paper)
__Standard oxidizer - potassium dichromate 5%
__Ascorbic acid, solid

7.0 HEALTH AND SAFETY PRECAUTIONS:

7.1 Minimum Personnel Protective Equipment (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 6.1.2) 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.

9.6 Record in the Field Log Book/Field Notes that the sample was treated for oxidizer or that the oxidizer test was negative. Also enter information on the appropriate data sheet.

10.0QUALITY CONTROL

10.1KI starch strips are checked for functionality prior to use.

11.0REFERENCES

11.1SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Update IIA, September 1994. Method 9010.

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NAVY ENVIRONMENTAL SAMPLING AND FIELD TESTING PROCEDURE

STANDARD OPERATING PROCEDURE

FIELD TEMPERATURE DETERMINATION

PREP BY:
APPROVED BY:
EFFECTIVE DATE:

CAUTION

This Standard Operating Procedure has been prepared as an example to this manual 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.

1.0 TITLE: SOP 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 one (1) degree).

5.2 Rinse waste water container.

5.3 ASTM Type I or II water squirt bottle.

5.4 Clean wipes.

5.5 Rubber gloves.

5.6 Field Log Book/Field Notes.

5.7 Personnel Protective Equipment (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 Personnel Protective Equipment (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 Field Log Book/Field Notes 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.

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 (NBS) - traceable certified thermometer.

9.3 The calibration of dial - type thermometers shall be checked at least quarterly against a NIST (NBS) - traceable thermometer.

9.4 Do not use thermometers where the indicating fluid has become separated.

10.0REFERENCE:

10.1Standard Methods for Examination of Water and Wastewater, 19th Edition,: Section 2550 B, Laboratory and Field Methods.

FIELD TEMPERATURE DATA SHEET

Thermometer Control Number: ____________

Calibration Expiration Date: ______________

Analyst: ______________________________

Remarks, Conditions: ____
____
____

Temperature Log

Date/Time	*Sample Location*	*Reading ℉ / ℃*	*Analyst Initial*	*Remarks*

Reviewed By: ____________

Date: _________________

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NAVY 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 to this manual 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.

1.0 TITLE: Calibration & maintenance of a Photovac Photo Ionization Detector

2.0 SCOPE AND APPLICATION

2.1 To calibrate and perform periodic maintenance on a PhotoVac Photo Ionization Detector (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.

** Recalibration must be performed after each maintenance operation.*

4.0 EQUIPMENT

4.1 Obtain the following equipment:

- *Photo Ionization Detector (PID)*
- *Manufacturer's Instructions*
- *Spare battery*
- *Battery charger*
- *Spare bulb*
- *Spare particulate filter*
- *Wrenches, screwdrivers, etc., as required, to disassemble the instrument for maintenance*
- *Cotton tipped swabs*
- *Clean/New Sampling bags*
- *Field Log Book*
- *Pen*

5.0 REAGENTS AND MATERIALS:

- *Methanol*
- *Cylinder of pure air, or activated carbon filter to make pure air and a sampling pump to fill the sampling bag*
- *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 which 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.

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 Field Log Book.

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 two minutes to come to equilibrium before calibrating.

8.6 Fill the bag with clean air (either "Zero Air" from a cylinder or air which 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 Field Log Book. 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 Field Log Book.

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 Field Log Book. 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 Field Log Book.

CAUTION

Certain maintenance procedures may be performed in the field, but 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 micron cartridge-style filter designed to capture particles which 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 5 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 Field Log Book.

CAUTION
Certain maintenance procedures may be performed in the field,
but 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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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

CHAPTER 15

GUIDELINES FOR REQUESTING LABORATORY TESTING

15.1 PURPOSE. This chapter provides guidance on requesting laboratory testing for compliance sampling. When requesting laboratory testing the following should be addressed:

- Responsibility of all parties
- Parameter Selection
- Method Selection
- Laboratory Selection
- Turn around Time
- Quality Assurance/Quality Control (QA/QC)

15.2 SCOPE. Communication with the laboratory prior to, during, and after sampling is necessary to ensure the data reported will meet the regulatory requirements. The extent and information communicated to the laboratory is dependent on the data needs, the laboratory expertise, and the stringency for adherence to the testing requirements. **Appendix G** provides contact information for many of the Navy and Marine Corps multiservice laboratories.

The parameter, test method, holding times, sampling method, sampling frequency, preservation, quality control, and reporting are prescribed by the permit or regulation and must be adhered to for all data reported. This information must be communicated to the laboratory to ensure that the data generated is usable for compliance purposes.

15.3 HAZARDS AND SAFETY PRECAUTIONS. See **Chapter 3, Section 3.3.3** and **Chapter 12, Section 12.3.3** for restrictions pertinent to explosive samples. See **Chapter 4, Section 4.6** for Department of Transportation (DOT) requirements for shipping samples to the laboratory. Samples must be properly labeled, and the outside container properly marked, to be sure the laboratory receiving the samples complies with all hazardous material regulations while opening and handling the shipping package and containers. The laboratory's hazard communication program must be in compliance with 29 CFR 1910.1200 to ensure proper handling during sample custody, transfer to the laboratory, sample analysis, and storage.

15.4 PREPARATION. The general steps for requesting laboratory testing are:

1. Determine the reason for testing: regulation required testing, permit application, permit monitoring, material characterization, process control, treatment design, research, site study, problem solving.
2. Select parameters required for testing.
3. Determine levels of measurement expected and levels of detection required (i.e. per cent, parts per million, parts per billion, or sub parts per billion).
4. Determine matrix to be monitored, i.e. water, soil, waste, air, oil or multiphasic material.
5. Select method(s) based on parameters, detection levels, matrix, and regulatory requirements.
6. Review method precision, accuracy, and performance for matrix and regulation. If method performance is not presented in the method, a matrix specific study may be needed to evaluate method performance for the matrix being measured, at the level of detection expected, and the required detection limit.
7. Review the Field Sampling Plan (FSP) to ensure sufficient sample, preservative, and bottles are provided for the methods selected. Ensure needed information and materials are forwarded to the laboratory.
8. Develop QC requirements for the project based on measurement objectives and regulatory requirements.
9. Select the laboratory that demonstrates acceptable performance with methods, matrix, levels of detection required, in-house control charts, and analyst proficiency records. The laboratory should have the necessary accreditation, certification or approval, and proficiency testing.
10. Finalize the Quality Assurance Plan (QAP) for the project based on final input from the sampling team, laboratory(ies), and regulator(s). Develop a time line based on sampling time, turn around time, and project needs.

NOTE:

These steps do not include contracting requirements but relate only to the technical aspects of requesting laboratory testing services. See **Appendix L** for suggested text for use in contracting laboratory services.

15.5 RESPONSIBILITY OF ALL PARTIES. The permit holder or generator, sampler, and testing laboratory are responsible for proper performance of the protocols associated with the regulated activity. The role of each party involved with the sampling and testing should be clearly identified and lines of responsibility agreed upon prior to initiation of the sampling event.

It is the ultimate responsibility of the permit holder, generator, or regulated party to obtain valid test data. This includes proper sampling design, collection, safety practices, requesting the correct test method and quality control from an analytical laboratory. For compliance sampling, the choices of sampling site, sampling methods, sample amounts, preservation, holding times, and report format are prescribed by the regulation. Whether the regulation is Federal, state administered, or permit driven, the parameters, levels of concern, and quality control are specified and must be communicated to the laboratory to ensure that the data generated is legally reportable and defensible. The permit holder is responsible for balancing the amount of QC required and the degree of risk associated with data defensibility. In some areas of the country, the information needed for reporting and quality control are not part of the program requirements.

Sampling personnel are responsible for:

- Adherence to sampling and preservation procedures
- Sample collection in adherence to the permit or regulation
- Locating the correct sample location
- Label samples for proper identification by others
- Documentation of any deviations to sampling procedures
- Recording all field observations
- Initiating or maintaining custody records
- Sample preservation
- Notifying the laboratory of expected sample arrival date/times as necessary
- Timely delivery of the samples to the laboratory to ensure sample integrity

The laboratory is responsible for:

- Providing prepared containers for sample collection
- Performing the analysis as requested by the customer
- Reviewing all data and reporting the requested information within the **turnaround time (TAT)**
- Preservation checks
- Advising the customer of any problems, deviating from protocols, or control criteria performance
- Providing technical assistance, if requested, for data interpretation

The laboratory must be aware of required levels of detection, acceptance criteria for duplicates, spikes, blanks and calibration checks (where applicable), and when method performance criteria must be better than the referenced method requirements for the compliance samples collected.

15.6 PARAMETER SELECTION. The parameter selected for analysis is found in the applicable permit, regulation, or other compliance document. The most common compliance situations are presented as examples:

National Pollutant Discharge Elimination System (NPDES). In the NPDES permit Application, Form 2C, all parameters controlled by the regulation are listed in the application. Testing parameters are selected from the listed parameters and are based on industrial category, known present and absent chemicals, and **conventional pollutants**. The application lists the sampling methods to be used for each group of parameters. After permit review by the regulator, ongoing monitoring is stipulated in a discharge permit. Wastewater monitoring for NPDES permits list the test, frequency of testing, method of sampling, level for compliance, and QC samples. The approved test methods for each parameter, holding times and preservations are listed in 40 CFR Part 136. See **Appendix H** for examples of NPDES wastewater program requirements.

Public Owned Treatment Works (POTW). Discharges of municipal wastewater to public owned treatment works (POTW), may require local permits for discharge. The type of treatment plant and loading capacities along with the industry category will be used by local regulators to determine initial monitoring for permit application. Routine testing is performed based on the applications data, process knowledge and review of on-going test data. Similar to the NPDES permit, test, frequency, sample location, method of sampling and QC samples will be described in the permit. The approved test methods for each parameter, holding times, and preservations are listed in 40 CFR Part 136 and are similar if not identical to NPDES wastewater requirements.

Safe Drinking Water Act (SDWA). Drinking water testing is performed based on Federal state, and local requirements. The parameter, test method, frequency of testing, method of sampling and level for compliance is found in the operating permit of the water supply, along with any additional updates to the regulations. The method dictates the QC and sampling considerations for preservation, handling and storage. **Chapter 9** and **Appendix H** provide details on sampling methods and parameters selected for testing. Parameters, frequency of sampling, sampling locations, compliance levels, method references, and reporting requirements are found in 40 CFR Part 141.

Waste. For characterizing a waste as hazardous, the complete regulations found in 40 CFR Part 260 to 261 must be understood before deciding on the tests to be performed. It is not always required by regulation to test a waste to determine if it is hazardous. User knowledge of waste may be used to determine proper disposal requirements. However, this practice would not pickup a change in the process which generated the waste and possibly the waste characteristics. Wastes are classified as hazardous if they are specifically listed in 40 CFR Part 261, are deemed hazardous by the generator, or meet the characteristics of a hazardous waste as specified in 40 CFR Part 261. The hazardous waste generator is responsible for characterizing his waste. The generator prepares, signs, and is liable for a manifest that accompanies the hazardous waste to the treatment storage, or disposal site. The waste transporter is bound by the information on the manifest. The transporter does not typically sample or analyze the waste. The owner or operator of the treatment, storage, or disposal facility receives a copy of the manifest signed by the generator (and the transporter) and returns a signed copy to the generator. He may or may not sample and analyze the waste prior to treatment or disposal. As an example, when incinerating waste material, the amount of metals, chlorinated compounds, Polychlorinated Biphenyls (PCBs), and other materials are needed so the treatment and/or disposal firm may assess the waste material and potential air contaminants it will generate during handling of the waste.

For regulatory compliance, the waste generator must analyze the waste to ensure that the methods and quality controls used are acceptable for regulatory reporting. The laboratory may be a Navy laboratory or contracted laboratory. Data from the contracted waste hauler is of limited value for regulatory reporting by the waste generator.

Sediment. Sediment sampling is most often performed when dredging permits are being requested from the regulating body. Permit applications vary depending on stream use, local concerns, and known contaminants. Sediment parameters sometimes include characteristics of a hazardous waste, PCB, dioxin and furans, and all NPDES parameters. Both the surface water and the sediment may be required for monitoring. The sampling is dependent upon the dredging method to be used and based on local regulatory review of the permit application. The sampling and testing parameters must be reviewed with the regulations before starting any sediment sampling and testing.

Groundwater. Groundwater sampling may be performed for drinking water testing or for landfill monitoring at both hazardous or solid waste landfills. For drinking water testing, the parameters and quality control are based on the drinking water regulations found in 40 CFR Part 141. For landfill monitoring, the sampling and parameters are performed based on the permit developed during initial landfill development and permitting. The permits include sampling method, frequency, filtered or unfiltered samples, purge method, levels for compliance, and test parameters. The requirements of 40 CFR Part 265.90, Subpart F - Ground Water Monitoring should be reviewed for landfill ground water regulations. The regulations for landfill leachate monitoring are found in the solid waste landfill regulation in 40 CFR Part 258.

Clean Air Act (CAA). Air monitoring may be required for a permit application, permit compliance testing, or personal protection. All parameters, frequency of testing, and test methods are found in 40 CFR Part 60. A complete discussion of air monitoring is found in **Chapter 10**. The parameters to be monitored are selected based on discussions with state regulators and stipulated in the operating permit. Personal monitoring is regulated by OSHA and is not EPA controlled.

In all environmental monitoring, it is of paramount importance to know and evaluate your permit and regulatory requirements during application, negotiation, and evaluation with the regulator. The parameters tested should be limited only to those required and not go beyond the scope of the permit. For example, having the lab report all the EPA 624, Gas Chromatography / Mass Spectrometry (GC/MS), volatile organics on a report for NPDES compliance is not necessary when only benzene and toluene are required. Since these compounds are known to be present, a low detection limit, and confirmation of the parameters identity is not required, it may be less expensive and acceptable to perform the benzene and toluene by EPA method 602. This is a Gas Chromatography (GC) method which usually provides faster turnaround at a lower cost. A second example is during waste characterization. If the waste is known to be from a metal plating area and the only materials used in the process are metals, there is no regulatory need to perform organic testing. In waste classification, it is perfectly acceptable to use generator knowledge for classifying and selecting the parameters.

NOTE:

> In hazardous waste classification, if the generator knows the waste contains organic material and the results indicate no organic material was found, the generator must be extremely careful in declaring the waste non-hazardous, since matrix and method variability may result in data that is susceptible to false negatives.

15.7 METHOD SELECTION. The selection of the correct test method is necessary for compliance and for legally defensible data. After selecting the parameters to be monitored, the methods must be chosen to assure the legally required method is performed and the level of compliance and detection will be per the permit or regulated requirements.

Many methods exist that are scientifically defensible and useable for environmental studies, research, process control, and process design. These methods must not be confused with compliance methods. The data from non-compliance methods should be clearly identified so the user of the data does not misinterpret data collected for other reasons. Data to be used as legally defensible must be performed according to the methods listed in the law, by the regulator, or in the permit.

The methods are to be performed as written. Modifications to the methods are allowed only if no change in chemistry results. The laboratory professional may use discretion in some steps of the method where the method indicates "or equivalent" such as; selecting instrument conditions in order to optimize the resulting data. However, the exact method used and any deviations must be documented exactly as performed to ensure repeatability of the method.

In no case, is the method performance, as stated in the legally established method, allowed to be compromised. Method performance, as measured by precision and accuracy, detection limits, and the required method quality control data must not be degraded. Whenever modified methods are used by a laboratory, records must be maintained to demonstrate that the modified method produces the same results as the method referenced. In some cases modified methods are needed when matrix affects do not allow the data produced from the matrix to meet the method performance criteria. These modifications must be reviewed with the regulating body prior to, during, and after the modification development.

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.

One of the concerns when selecting methods is the level of measurement or the **method detection limit (MDL)**. Certain equipment and methods only perform within a certain concentration range and can measure only to a certain limit. The detection limit is the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability. Legally defensible data requires a calibration curve or proof of linearity for the method performed. This means that reference standards traceable to national standards must be performed that bracket the measurement range (high, middle, low). When single standards are performed to verify calibration range, the standard is either the reporting limit (low) or the middle standard depending on the method requirements. Where method requirements do not exist the lab selects the calibration verification which may be high, middle, or low depending on the labs QC requirements.

As an example of selecting a method based on level of detection, atomic absorption is an acceptable technique when analyzing for metals from 0.1 to 100 parts per million (mg/L or mg/kg). This range is linear for most elements and does not require the analyst to dilute the sample. Diluting the sample may add error to the final results, depending on the amount of dilution needed. If results are needed below 0.1 ppm or less than 100 parts per billion, more sensitive techniques such as Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) are available that are legally defensible for drinking water testing. For other compliance testing, Inductively Coupled Plasma (ICP), with a sonic nebulizer or horizontal torch is needed to reach these limits for most metals.

The MDL listed in most EPA methods refers to the detection limit expected under standard laboratory conditions when testing laboratory ASTM Type I or II water. The MDL is commonly accepted to the limit calculated using 40 CFR Part 136, Appendix B in laboratory ASTM Type I or II matrix. The MDL is the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, qualitatively or quantitatively measured, and reported to be greater than zero. Some EPA methods refer to detection or measurement limits, such as practical quantitation limits (PQLs), quantitation limits, or levels of detection when referring to a value above the MDL. These measurement limits are often based on the MDL and by further statistical calculation or trial and error. Uniform standards and criteria regarding the detection limit, reporting limit and method detection limit are still being developed. When selecting any method, additional quality control may be necessary to verify the detection limit or reporting limit is valid for the matrix being tested.

Each lab will develop a calibration curve and acceptance criteria within the level of detection most often performed by the laboratory. Differences in concentration ranges may result in slight differences (less than 20%) in reported values from one laboratory to another. If these differences need to be controlled, the exact method must be known. When split sampling is performed, the identical method must be performed by each laboratory.

Test methods contain the documentation describing the required quality control elements. The newer EPA methods all follow the same format. This format is referred to as the Environmental Monitoring Management Council (EMMC) format. This is the format agreed to by all EPA agencies for writing methods. The format includes the following:

- Scope and Application
- Summary of Method
- Definitions
- Interferences
- Safety
- Equipment and Instrumentation
- Reagents and Supplies
- Sample Collection, Preservation and Storage
- Quality Control
- Calibration and Standardization
- Procedure
- Data Analysis and Calculations
- Method Performance
- Pollution Prevention
- Waste Management
- References

The above elements are required for ensuring comparability of laboratory method performance. The regulated community has the responsibility to assure that the test data submitted contains the required elements. Otherwise, the data can be considered noncompliant or not legally defensible.

For EPA method selection, five categories exist:

Drinking Water. 40 CFR Part 141.21-142.30 and "Manual for the Certification of Laboratories Analyzing Drinking Water" (April 1990). Methods for organic testing are 500 series methods, inorganics are 100 to 400 series methods. Other methods include: Standard Methods for the Examination of Water and Wastewater, and ASTM, Water and Environmental Technology, methods.

Wastewater. 40 CFR Part 136; EPA Organics methods are 600 series and inorganics are 100 to 400 series methods.

Groundwater at Solid Waste Landfills. 40 CFR Part 258.50; Appendix I & II refer to SW-846 methods for analysis of Solid Waste.

Solid Waste & Hazardous Waste. 40 CFR Part 261; Methods manual is SW-846, Test Methods for Evaluating Solid Waste Physical Chemical Methods, Updates I, II, IIA and IIB. Methods are numbered from 1000 to 9999. The latest water methods available for draft review are not currently promulgated. These methods are numbered as 1600's and must not be confused with SW-846 methods.

Underground Storage Tanks. 40 CFR Part 280; The methods for removal and additional monitoring are specified by state agency for soil and groundwater monitoring.

15.8 LABORATORY SELECTION. The following should be considered when selecting a laboratory for compliance testing:

- Certification/Accreditation/Approval
- Analytical methodology
- Quality control
- Reporting
- Auditing, internal and external
- Turn around time
- Cost
- Geographic and time constraints

15.8.1 Certification/Accreditation/Approvals. Laboratory certification programs are part of the drinking water regulatory program requirements. Therefore, all drinking water compliance samples must be tested by laboratories certified by the agency with primacy for drinking water. Some states also require certification for wastewater testing and hazardous waste testing. All certification, accreditation, and approval programs require:

- On-site inspection
- Proficiency testing or performance evaluation testing

15.8.1.1 **<u>On-Site Evaluations</u>.** During the on-site inspection, the auditor reviews the operations of the laboratory to ensure the equipment, facilities, and staff are available and adequate to perform the testing. The laboratory must have the following:

- Adequate facilities/equipment
- Staff
- Reagents of adequate quality
- Standards and reference materials traceable to the **National Institute of Standards (NIST)**
- Documented methods
- Standard operating procedures
- Quality assurance plan

The laboratory must have personnel with educational backgrounds and continuing training suitable for their assigned tasks. Documentation of capabilities must be maintained. Laboratory management must provide maintained facilities, well lit, adequate heating/ventilation/air conditioning, work and storage space, and waste handling facilities. A Chemical Hygiene Plan and a Right to Know Plan must be documented and available. Laboratory management must also provide for sample receiving, sample storage, calibrating equipment, and calibration supplies. These may be standards traceable to the **National Institute of Standards and Technology (NIST)** and purchased standards of known composition. Calibrated weights and balances, calibrated thermometers, spectral wavelength filters, and other standards as required for the tasks of the laboratory must be evident. The laboratory must provide policies on subcontracting of testing or calibration, quality of outside suppliers, and how external and internal complaints are handled.

The certification, accreditation, approval is granted by state agency, Federal agency, or other entity and accompanied by a list of parameters, methods, or matrices. This is referred to as the **Scope of Accreditation or Certification, or Approval**.

Requiring a laboratory to be a **Contract Laboratory Program (CLP) participant** is only required when performing work for CERCLA or other remediation investigation as required by Federal or state law. CLP is the program established by the EPA as a contractual program for performing testing for the EPA in cases of litigation with responsible parties. The **Statements of Work (SOW)** are different for each contract released. The number of CLP labs in the country vary depending on EPA contracting needs and do not reflect the laboratories ability to produce scientifically defensible data.

A CLP report package is an EPA format for reporting data that is usually not be required by the regulatory authority for compliance purposes. Requesting a CLP package may add unnecessary cost to the compliance project with no additional assurance of data suitability. The purpose for CLP testing, package, and validation are different than routine compliance monitoring.

Do not over qualify or misqualify laboratory selection criteria. The methods, matrix affects, and level of performance differ with each program. Requiring a lab to be qualified in a program that is beyond what is required, will not produce more reliable data, it may only add to the price and turnaround time.

The Navy approval program reviews a laboratory's facilities, equipment, and staff to ensure proper performance of the requested parameters and methods. This approval and the on-going laboratory demonstration of control criteria should be used in lab selection for technical acceptability. The following items are part of most approval, certification, and accreditation programs.

15.8.1.2 **<u>Proficiency</u>.** Another way to assess the reliability of a laboratory is to review **performance evaluation (PE) results**, sometimes called **proficiency testing (PT) results**. Samples are furnished by certifying or accrediting agencies which contain known amounts of parameters in different matrices. The analytical results are evaluated by the agency for accuracy using a statistically derived criteria. These samples indicate how well a laboratory can perform against its peers. Two examples of performance evaluation samples are: the EPA samples for the drinking water supply program, and wastewater discharge PE samples for NPDES permit compliance. Some states provide samples for their state certification process. Proficiency testing is also conducted by other Federal agencies and industries using commercial suppliers of reference materials. Requesting the labs PE or PT data for a given matrix and method will assist with the selection of the lab. At least two rounds of testing should be provided to review on-going demonstration of proficiency.

15.8.2 **<u>Analytical Methodology</u>.** The laboratory performing the work may refer to methods by the EPA criteria or refer to in-house procedures based on the regulated method. The scope and procedures performed by the lab should identify the applicable regulatory program, matrix, and reference methods. Caution must be exercised when using the labs' catalog for selecting methods. The catalog may reflect acceptable methods for the area in which the lab is located and may not be applicable to the compliance requirements at the site's location. Always ensure the laboratories methods are acceptable to the regulator. Lab methods may not be updated as quickly as the regulations.

Some labs have customer service or technical representatives that assist users of the lab services on applicable methods, sampling considerations, and other QC information. These services are usually provided at no additional charge and the quality of these services is dependent upon the individual experience and training of the laboratory. Method selection based on the recommendation of the lab must be verified to ensure the correct methods and to maintain legally defensible data for the measurement objectives.

15.8.3 Quality Control. All measurement objectives have at least two aspects to quality control, the lab QC and the project QC. When selecting a lab, the QC performed by the lab on a routine basis must be weighed with the project's QC objectives. All labs run QC control samples made in ASTM Type I or II water with each batch of samples. This data is statistically plotted to observe trends on the control samples. Control criteria (accept/reject) may be established based on these control samples. The labs in-house control criteria must be at least as stringent as the regulated method, but may be better than the requirements. Requesting the lab's control data for the method and matrix of interest, will provide the user with a report of the lab's ability to control the method, the frequency of using the method, and the acceptance criteria.

Other quality controls that may need to be assessed when selecting a lab for "clean" measurements or low level testing, are blanks and reporting limits. The establishment of reporting limits (in comparison to the method detection limit), is of concern when false positive and negatives near the reporting limits will result in compliance failures. Results of the laboratories' analysis of blank samples will indicate how clean and contaminant free the laboratories' facilities and instruments are kept. High values in the analysis of a blank sample can indicate cross contamination of samples in storage, carryover from previous samples analyzed on the instrument, or poor housekeeping.

15.8.4 Reporting. Environmental laboratories provide monitoring information used to demonstrate compliance with various laws. To meet these requirements, the information must be legally defensible. If the regulatory agency challenges the report and sufficient required documentation cannot be produced, the report is indefensible and the generator or permit holder may be subject to fines.

When capacity is exceeded, or method performance problems prevent the laboratory from performing a test, the laboratory may subcontract for that analysis unless specifically required not to allow subcontracting. Subcontracting to multiple laboratories may be needed for some compliance programs when a variety of matrices and parameters are requested. In all cases, when subcontractors are used for producing data, they should be identified on the final report from the lab.

The laboratory must be able to prove that customer results are kept absolutely confidential during processing, any electronic transmission, and during reporting. Records of raw data and final reported data should be retained and readily retrievable.

The raw data required for final data interpretation shall be available for sample analyses and calibrations, verifications blanks, matrix spikes and duplicates, and other QC data required by the method. Record retention should be clearly indicated in the QAP and made known to all parties as to time and extent of the records to be retained. The length of time for record retention is laboratory-dependent and should be a minimum of three years, unless otherwise specified in the regulation.

15.8.5 Auditing. Laboratory auditing is performed by external agencies on a routine basis to ensure that the agencies' program criteria are being met. In addition to external auditing, the laboratory should have a process for self auditing when problems are found either by lab employees, customers, or regulatory agencies.

Every laboratory receives complaints about sample results. The laboratory must have a system to handle, correct, and improve its Quality System. The investigation and resolution of complaints or corrective actions should be implemented and documented. The tone of and seriousness about a laboratory's complaint resolution system, or lack of it, will tell much about the laboratory's quality assurance program.

15.8.6 Cost. Laboratories operate with large fixed costs for equipment, facilities, supplies, and personnel. Unreasonably low prices for analysis should always raise suspicions of a method shortcut, incomplete QC, or lack of data documentation. Prices are normally quoted for sampling, shipping, preparation, analysis, special reporting, and any requirements for expedited turnaround time.

The most important objective in compliance monitoring is the quality control and data traceability of the sample and internal QC of the lab. Purchasers of lab services must be willing to pay a sufficient amount to ensure that the QC, traceability records, and personnel are available years after the sampling and testing have been completed. For compliance monitoring, emphasis should be put on laboratory accreditation, certification, approval and reliability, and not on economy, since fines, permit violations, and congressional investigations may result from insufficient records or Quality Control.

15.8.7 Geographic and Time Constraints. In selecting a laboratory, consideration must be given to the location of the laboratory in proximity to the sampling location. This becomes necessary when collecting samples with short holding times of 24 hours or less. Coordination with the laboratory to ensure that the staff and resources to handle the sample immediately upon arrival must be part of the selection criteria. When holding times are 24 to 48 hours, more remote laboratories may be used with overnight shipping to send the samples to the laboratory. This additional cost must be considered, and the labs ability to process the sample upon receipt must be assured so holding times will be met. Repeatedly missed holding times are an indication the lab does not have the resources and staff needed to meet the requirements. Repeatedly missed shipping times may also be due to the courier not having sufficient resources to meet the requirements.

When shipping from remote locations during unusually hot or extreme cold conditions, sample handling may require unique procedures to assure arrival at the lab without freezing or overheating. In some cases only refrigeration units are suitable. If it can be demonstrated that longer holding times, or temperature effects do not affect the data quality, the regulator may allow a variance for special conditions. This variance must be obtained in writing in advance of the sampling event.

Unusual or emergency situations may arise from time to time which require additional testing in considerably less time than standard lab turnaround. The lab selected for the routine testing should be used for emergency testing, to assure data comparability. The lab's performance in emergency situations should also be part of the selection process.

15.9 TURNAROUND TIME. When selecting a laboratory, (and delivery dates are critical), the lab should be able to produce some form of information on typical turnaround. A laboratory should be able to produce a report of promised versus actual turnaround times for a specific test or method.

15.9.1 Regulatory Holding Time. The time from sample collection until the start of the test analysis is the **holding time** referred to in the regulation. The holding time was developed by the EPA for a number reasons. One reason is chemical stability and uniformity of method start times. Some chemicals are not stable for long periods of time in the matrix. The EPA recognized the need for all samples to be analyzed within a given period of time to ensure comparability between laboratories. Some chemicals and matrices may have less or more stability than the EPA accepted holding time criteria. Variances are sometimes requested as part of the permit negotiations to modify holding time requirements based on method and matrix specific studies. In all compliance reports, the holding times published in the regulation, or modified in a permit, must be met to produce data that is legally defensible. Samples must be received by the laboratory and the laboratory must start the preparation or analysis within the required holding times.

15.9.2 Laboratory Analysis Time. Turn around time is defined by laboratories as the time it takes from sample receipt until data is reported to the customer in the form of the final report. The turnaround time includes, as necessary, time for sample receiving, preservation checks, sample preparation, sample analysis, data calculation, data review by analyst, second reviewer, quality assurance review, report preparer, and lab manager. In general, a two (2) week turn around time is typical and reasonable for any analysis.

The best way to determine turnaround time is to ask the laboratory. However, if time estimates are needed prior to selection of the laboratory, the following is presented to assist with allotting sufficient time to receive results from the lab test. Most compliance testing for the standard, wet chemistry, metals and organics on water, wastewater, and soil is performed within two weeks of sample receipt. A number of laboratories provide services for less than two week turnaround at special fees or for a limited number of parameters and matrices. Waste characteristics, oils, and complex matrices or non-routine parameters may take one to two months to provide acceptable data.

During method selection, the time required for an analysis may be estimated based on preparation and analysis time given in the method. Sample preparation steps may be required as part of the method or may be an addition to the method in order to prepare a matrix for the sample analysis. Preparation steps may take from 15 minutes to 24 hours. Sample analysis may take from one hour to seven days to perform. Even the less cumbersome methods, where direct readout of final results is recorded, time must be allowed for calibration, data review, quality control review and entry, and final data reporting and review.

Most laboratories require at least one day for quality control review and reporting. Less than one day is possible through 24 hour operations, automated systems, and automated review processes in the laboratory. However not all methods are automated and therefore time for the administrative and quality assurance functions must be estimated.

In addition to administrative and QA requirements within the lab, work load contributes to the turnaround time of the reported results. Laboratories capable of handling one to twenty samples of a given analysis may not be able to handle one hundred samples for that test within the same time frame. Conversely, labs capable of handling fifty samples per lot may not be as responsive to sample lots of less than ten. These factors will contribute to turnaround time.

For the most common compliance analyses, **Table 15-1** lists the minimum time needed for analysis of lots of less than ten samples. In general, add one day for sample receiving, preservation check, and custody review at the start of processing and one day at the end for data and QC review by the lab. One day equals approximately a ten hour processing day. Laboratories performing wet chemistry, instrumental analysis, and staffing 24 hours per day may provide faster service in processing and QC review. They cannot, however, offer a shorter analysis time if they are performing the method as written.

When quality control or instrument performance in a given batch does not meet internal acceptance criteria, sample rerun may be required where holding times allow. Sample rerun may include preparation from the original sample or reanalysis of the prepared sample, depending on the information provided by the control samples.

When ordering expedited TAT for emergency compliance situations, you are paying the lab to not hold the sample and expedite the QA review. In no case can a compliance sample have a run time less than the method required analysis time, since the regulated method must be followed as stated for legally reportable data. Method modifications are only acceptable if the state or local agency provides written acceptance of the modified method.

15.10 QUALITY ASSURANCE/QUALITY CONTROL. When developing the project for compliance monitoring, the objectives, data comparability, and validation should be addressed.

15.10.1 Measurement Objectives. Data Quality Objective is a term commonly associated with remedial action tasks or waste characterizations. It is a process whereby information on an environmental problem, real or perceived, is refined until a clear and unambiguous statement of the problem and possible solutions can be made. A team of stakeholders, decision makers, and technical support personnel organize and evaluate the information.

Compliance objectives are concerned with providing regulators with legally defensible data demonstrating that provisions of a permit or regulation are being met. The permit is the regulators' solution to the problem of protecting and improving the quality of the environment based on the laws passed by the lawmakers. The technical specialists in compliance efforts are sampling personnel and the environmental laboratory which function under quality control and quality assurance dictates. All operations are performed under the dictate of regulation and a QA scheme to ensure comparability with the regulators sampling and lab teams, as well as other potential litigator's sampling and lab teams. All efforts are made to assure representative sampling and data comparability that will stand up to the rigorous scrutiny from both the scientific and legal communities.

For this reason all QC actions are specified in EPA methodology. For example, the 500, 600, 1600 and SW-846 methods for compliance monitoring have an entire section on required QC. In addition to method specified QC for the laboratory, field operations must include QC of the sampling event and validation that the methods selected are acceptable for the matrix being sampled. When compliance problems arise, assurance of method performance due to matrix effects, sampling variability, method performance and laboratory performance are examined. Additional QC samples may be collected and more rigorous sampling protocols may be instituted for short periods of time.

Table 15-1 Approximate Turnaround Time*

Parameter	Preparation Time	Analysis Time
BOD	0.5 day	5 days
Solids	0.5 day	1 day
Wet Chemistry	1 day if required	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

- Add one day at the beginning for sample receiving and one day at the end for QC review and reporting.

Improvements to treatment or system design may require additional monitoring for process control, requiring less rigorous QC protocols. All samples are collected from the same location, but the purpose of the testing will dictate the QC and detail required for the sampling event. Consideration must be given to each of the following and should be included in the Quality Assurance Plan for the compliance monitoring project:

- Organization and management
- Quality system audit and management review
- Personnel and training
- Accommodations and environment
- Equipment and reference materials
- Measurement traceability and calibration
- Test methods
- Handling of test items, records, reports
- Subcontracting of testing
- Outside support and supplies
- Complaints handling and corrective actions

15.10.2 Data Comparability. Laboratories can demonstrate their ability to obtain comparable data through the use of performance evaluation samples (PE samples) or round robin samples. In some cases comparison (PE samples) data is required with the annual QA report or lab certification.

For example, NPDES PE samples are obtained from state certifying agencies twice per year. Successful analysis must be accomplished to retain approval from the permit holder for performing the sample analysis.

When data comparability is critical to compliance reporting, the permit holder may require split samples between one or more approved labs to ensure data comparability. As part of the QAP, the acceptance criteria for split samples is agreed upon and when data is not within this acceptance criteria, the sampling, methods and detailed data review is conducted.

15.10.3 Validation. In most compliance reporting, no independent review of the lab data is performed, unless compliance limits are exceeded or unusual data results are reported by the laboratory. In these cases, the lab's ability to produce defensible data and enough information to reproduce the test conditions is critical for data review. Data must be organized so the analysis can be reconstructed.

To ensure analyst training is up to date, records should include a one time demonstration of proficiency of method performance using the method documented. On-going training is verified through the use of control samples and lab proficiency samples.

An independent reviewer should not rely on CLP data validation criteria for data review for compliance samples. The criteria may not meet the requirements of the compliance or method referenced. Data assessment should be made to prove accurate, traceable, and repeatable method performance. Any one or all of the following information may be required for validation when assessing compliance data:

- Sample identifiers, both client and lab
- Sample received logs, dates, times, and person
- Sample storage logs
- Extraction dates, times
- Analysis logs with dates, times, and person
- Run logs with dates, times, and person
- Sample volumes
- Preparation volumes, cleanup method
- Percent solids, moisture, if applicable
- Instrument/operating conditions
- Method reference
- In house SOP reference, with revision date
- Dilution data
- Confirmation
- Raw chromatograms, spectra data
- Intermediate quantitations
- Acceptance criteria for lab
- Acceptance criteria for method
- Data review
- Final reports

These parameters are assessed by the auditors for the various accreditation and certification programs. All of these should be made available for review and each item should be traceable back to any sample in question.

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CHAPTER 16

STANDARD OPERATING PROCEDURES

16.1 PURPOSE. This chapter provides guidelines for developing Standard Operating Procedures (SOPs) to assure consistency and standardization in sample collection and field testing.

16.2 SCOPE. Procedures to guide samplers and analysts in the field can be found in the Quality Assurance Plan (**QAP**), the Sampling and Analysis Plan (**SAP**), the Field Sampling Plan (**FSP**), the safety plan or Health and Safety Plan (**HASP**), as appropriate to the scope of the project, or Standard Operating Procedures. SOPs are the most basic and elemental of these documents, defining a discrete procedure and incorporating all the information a field sampler would need to properly take or analyze a sample. Field Sampling Plans can be written to include this Level of detail or SOPs can be incorporated by reference into the Field Sampling Plan.

SOPs can be prepared for all routine or repetitive tasks that are necessary for collecting samples and assessing environmental data. This may include, but not be limited to:

- Any administrative tasks associated with sample collection (i.e., who initiates sampling requests)
- Sample collection based on specific program, parameters, and matrix
- Instrument or method calibration
- Preventive/corrective maintenance
- Internal quality control procedures
- Site selection and sampling device placement
- Instrument/equipment selection, use, cleaning and decontamination
- Field measurements
- Sample management (preservation, hold time, shipping, receipt and handling, storage)
- Waste disposal

16.3 APPLICATION. Most sampling undertaken in accordance with permit or regulatory requirements has predetermined protocols. The sampling design, including location, number and size of samples, and order of collection, is determined by the regulation. Any considerations to the variability of the target analytes, the analytical method, constraints or access to the sampling area, and cost associated with sample collection must be explicitly granted by the regulating authority.

16.4 INTRODUCTION. SOPs are used to describe the performance of repetitive tasks and are usually written in a standard format. When following such a format and dealing with many variables, there are obviously going to be sections that are extensive or detailed for one process and minimal for another. The advantage of the standard format is that no subject is overlooked, and a quick assessment can be made as to which areas are of concern and which are not and, therefore, may be eliminated. The development of an effective SOP is important in ensuring the quality of work remains constant, and the job is performed correctly. However, one does not want the sampling SOP to be overly burdensome in areas of little concern.

16.5 FORMAT. The key is consistency. Choose a format that works for most areas and then conform to that as closely as possible for all similar applications. Not all elements may be required for all procedures. The following are suggested subject headings in the preferred sequence:

Title*
Document control information (on cover page)*
Scope and application*
Summary of Method
Interferences
Equipment
Reagents and Materials
Health and Safety precautions
Preparation of Equipment
Calibration and Standardization
Procedure*
Quality Control*
References*
Appendices
* denotes an element required of all SOPs

16.5.1 Title. The title should be concise but complete enough to identify the procedure, the material to which applies, and to distinguish it from similar titles.

16.5.2 Document Control Information. (Usually upper right corner of a Title Page) should include:

- Number of SOP
- Revision Number
- Date of Issue
- Page Number
- Name of authors
- Prepared for (name of organization)
- Approval signatures and dates
- Disclaimer (e.g. This Standard Operating Procedure has been prepared for the sole use of the Portsmouth Naval Shipyard and may not be specifically applicable to the activities of other persons or organizations)

16.5.3 Scope and Application. The scope and application of each procedure should briefly explain the specific purpose of the procedure and may include some information as to the type of samples for which the procedure was prepared, and any other limitations of the procedure. Indicate any training requirements and appropriate documentation for the procedure.

Include any warning needed in the interpretation of the results of the test.

16.5.4 Summary of Method. Include here a brief outline of the method, describing its essential features. A brief statement of the principles of the method may be given. State the type of procedure, such as colorimetric, electrometric, volumetric, etc.

16.5.5 Interferences. If the successful application of the procedure requires the inclusion of explanatory statements on interferences, include such information here. Interferences can be chemical for analysis or weather related for sampling.

List briefly the chemical constituents that are likely to cause interferences and the amounts that are known to interfere.

16.5.6 Equipment. Describe any necessary apparatus fully so it can be assembled prior to starting the procedure. Use Appendices for schematic drawings. Also list common laboratory apparatus such as flasks or beakers so all equipment may be assembled prior to initiation of procedure.

16.5.7 Reagents and Materials. List concentration, name of reagent, and procedure for preparing a fixed amount of solution. Indicate the requirement to use an analytical balance by writing grams with four significant figures after the decimal place. List only two significant figures if only an approximate quantity is required. If a standardization procedure is required, list it immediately after the procedure for preparing the solution.

Unless stated otherwise, all procedures shall use reagent grade chemicals and deionized/distilled water.

Include instructions to label all reagents according to the facility Quality Assurance/Quality Control (QA/QC) plan.

16.5.8 Health and Safety Precautions. Identify any significant health and safety concerns. Indicate personal protective equipment (**PPE**) usage, hazardous material handling precautions and appropriate training requirements.

Warning and Caution statements should be used in procedures prior to operating or maintenance procedures, practices, or conditions considered essential to the protection of personnel (warning) or equipment and property (caution). If proper personal protective equipment or clothing is not available, stop, and prepare for the situation. There is no excuse for taking risks where personnel injury, loss of life, or long-term health risks are possible.

16.5.9 Preparation of Equipment. Use this section when detailed instructions are required for the initial assembly, conditioning, or preparation for the procedure is required.

16.5.10 Calibration and Standardization. Give detailed instructions for calibration and adjustment of any apparatus as may be necessary to the use of the procedure.

16.5.11 Procedure. Include in sequence the detailed directions for performing the procedure. Group related operations into logical divisions. Use subheadings if they will help organize the material. Provide in detail the operations necessary to obtain required results. For example a sampling SOP should include such things as required documentation, sampling preservation, proper sampling technique, and equipment decontamination. Field testing SOP’s should include but not be limited to instrument parameters, analytical techniques, and required documentation.

16.5.12 Calculations. Provide directions for calculating the results of the test. Give a detailed equation for making each required calculation using capital letters for variables. Define each of the variables separately and avoid the use of combined factors. A typical calculation may be the best explanation for calculating results.

16.5.13 Quality Control. The statement of quality control provides users with a level of quality that is dependable, economic, and in conformance with specification requirements. Each method must specify how this level of quality is monitored and assured.

Strict adherence to the facility QA/QC Program must be addressed.

Examples of quality control measures which can be specified are:

- Review or oversight requirements
- Duplicate samples
- Percent recovery techniques
- Analysis of synthetic standards
- Control Charts
- Field blanks
- Acceptance and rejection criteria
- Corrective actions

16.5.14 References. List in alphanumeric sequence the designation number and complete title of all standards and specifications referenced in the procedure. Other analysts experiencing problems with a procedure, or wishing to expand it, may find additional information in the references. The following is the reference consulted in the preparation of this chapter:

Form and Style for ASTM Standards, 6th Edition, May 1983 ASTM, 1916 Race Street, Philadelphia PA 19103.

16.5.15 Appendices. Additional information may be included in one or more appendices to the method.

An Appendix is informative only, and is not a mandatory part of the procedure. Data forms for recording results, glossaries and material safety data sheets (MSDSs), may be included in Appendices.

16.6 DEVIATIONS. Any deviations from an SOP must be documented as to the justification and extent of the deviation, and how it may effect the applicability and legality of the procedure. Under specific circumstances deviations may require approval from supervisory personnel or the Program Manager.

16.7 ESTABLISHING THE NEED FOR AN SOP. The importance, nature, and repetitiveness of the activity or task will be factors to consider in requiring and developing SOPs. Management, technical staff, and the Quality Assurance/Quality Control Coordinator should identify tasks and activities for which SOPs are needed.

16.8 PREPARATION AND WRITING. Management will assign responsibility for preparation of an SOP. The person(s) preparing the SOP should be intimately familiar with the details of the activities/tasks involved and should consult with others who are knowledgeable and/or actively involved with the activities.

After selecting the format, reviewing all references and data that will form the basis for the SOP, it is time to develop the text. When writing each section, be concise and factual. If a glossary is necessary, it can be prepared for a compilation of SOPs, but reference notes should be provided individually where amplification or further explanations are warranted.

16.9 REVIEW AND APPROVAL. A draft SOP should be reviewed by at least one person intimately involved with or knowledgeable with the activity or task. Prior to final approval, the step-by-step procedural portion of the SOP should be validated by the reviewer through actual observation of the step-by-step activities. The final SOP is approved per the QA/QC plan. Because varied safety and health hazards may be encountered in the field or in the laboratory, all SOP's should be approved by the facility Safety and Health Coordinator. The SOP should also be approved by the QA/QC Coordinator.

16.10 FILING. Each SOP should be readily available as a reference in the work areas involved for those persons actually performing the task or activity. A complete master list and file of all SOPs should be maintained by the QA/QC Coordinator. The master file should include the original issue and all subsequent revisions.

16.11 REVISION. Revisions of the SOPs require review and approval, the same as the original SOP. To document revisions or changes follow the facility QA/QC plan for document control.

16.12 DISTRIBUTION. The original issue and the original of all subsequent revisions will be maintained in a master file. Copies of approved SOPs should be maintained in the office originating the SOP.

16.13 EXAMPLES. Examples of SOPs are provided in **Chapter 14.**

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APPENDIX A

BIBLIOGRAPHY OF EPA PUBLICATIONS

A.1 PREPARATION OF FIELD SAMPLING PLANS.

Data Quality Objectives for Remedial Response Activities. EPA/540/G-87-003. How to prepare a Quality Assurance Plan.

Data Quality Objectives for Remedial Response Activities: Example Scenario: RI/FS Activities at a Site with Contaminated Soils and Ground Water. EPA/540/G-87-004 (OSWER Directive 9355.0-7B). March 1987. A detailed discussion of how to select sampling locations and analytical methods. Appendix A is a list of EPA analytical methods and the demonstrated precision of each method.

Soil Sampling Quality Assurance User's Guide, 2nd edition. EPA 600/8-89/046, NTIS PB89-189864. 1989.

Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA. EPA 540/G-89/004. Oct. 1988.

A.2 SAMPLING PROCEDURES.

Ambient Air
Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air. EPA/600/S8-90/005. 1990.

Stack Emissions
Test Methods for Stack Emissions, 40 CFR 60 Appendix A. Definitive procedures for stack sampling.

Soil
Description and Sampling of Contaminated Soils - A Field Pocket Guide, EPA/625/12-91/002. November 1991. Chapter 3 is a good guide for the physical description of soils.

Characterization of Hazardous Waste Sites - A Methods Manual - Volume 2: Available Sampling Methods, 2nd edition. EPA 600/4-84-076, NTIS PB85-521596. 1984.

Groundwater
Guide to the Selection of Materials for Monitoring Well Construction and Ground-water Sampling. EPA-600/2-84-024, NTIS PB84-141779. 1984.

Practical Guide for Ground Water Sampling. EPA-600/2-85/104, NTIS PB86-137304. 1985.

Surface Water
Handbook for Sampling and Sample Preservation of Water and Wastewater. EPA-600/4-82-029. 1982.

Drinking Water
Pocket Sampling Guide for Operators of Small Water Systems. EPA/814-B-92-001.

Discharge Water
NPDES Compliance Sampling Manual. 1977.

Hazardous Waste
Samplers and Sampling Procedures for Hazardous Waste Streams. EPA/600/2-80/018, NTIS PB80-135353. 1980.

Test Methods for Evaluating Solid Wastes - Volume II Field Manual - Physical and Chemical Methods. SW-846 (NTIS PB88-239223 and PB89-148076). Sample collection procedures and detailed field and laboratory analytical methods.

A.3 CHEMICAL ANALYSIS PROCEDURES.

Field Testing
Test Methods for Evaluating Solid Wastes. SW-846 (NTIS PB88-239223 and PB89-148076). Both laboratory and field analytical methods.

Reagents, Solvents and Test Papers for Sampling. MIL-R-51197B.

Water - General
Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993. Revised analytical procedures for the laboratory measurement of turbidity, inorganic anions, cyanide, ammonia, nitrates, phosphate, sulfate, COD, and phenolics in water. Includes required sample containers, preservation, and holding time limits for each method.

Drinking Water

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Revised July 1991. *Supplement I:* EPA/600/4/90/020, July 1990. *Supplement II:* EPA-600/R-92/129, August 1992. *Method 525.2, Revision 1.0.* March 1994. Detailed laboratory procedures for drinking water. Specifies sample containers and sample volumes required for each method.

Waste Water

Laboratory Procedures: Analysis for Wastewater Treatment Plant Operators. EPA Region VII, Kansas City. Includes required sample containers, preservation and holding times for sewage samples.

Soil and Hazardous Waste

Test Methods for Evaluating Solid Wastes. SW-846 (NTIS PB88-239223 and PB89-148076). Both laboratory and field analytical methods.

Contract Laboratory Program (CLP) Statement of Work for Organic Analysis; CLP Statement of Work for Inorganic Analysis. Detailed laboratory procedures and quality control requirements specified by EPA for chemical analyses performed by contract laboratories under the EPA Contract Laboratory Program.

Air

40 CFR 60 Appendix A. Analytical methods to go with the specified stack sampling methods.

A.4 HEALTH AND SAFETY PROCEDURES.

29 CFR 1910.120. OSHA regulations for safety at hazardous waste sites, including required procedures and worker training and medical surveillance. Specifies requirements for health and safety plans.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities. DHHS (NIOSH) Publication 85-115, 1985.

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APPENDIX B

TRAINING SOURCES

The following information is provided to assist you in obtaining environmental training courses required to meet individual training needs:

NAVOSH and Environmental Training Center
9080 Breezy Point Crescent
Norfolk, VA 23511-3998
COMM: (804) 445-8778
DSN: 565-8778
FAX: (804) 445-8091

Commanding Officer
Naval School, Civil Engineer Corps Officers
Attn: C31C
3502 Goodspeed Street
Suite 1
Port Hueneme, CA 93043-4336
COMM: (805) 982-2895/2897
DSN: 551-2895/2897
FAX: 551-2918

Environmental Training Catalog
Naval Facilities Engineering Service Center
560 Center Drive
Port Hueneme, CA 93043-4328
COMM: (805)-982-5751
DSN: 551-5751
FAX: 551-4832

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APPENDIX C

HEALTH AND SAFETY PLAN REVIEW

The following information should be included in the site-specific health & safety plan (HASP). References are provided from OSHA and the Navy/Marine Corps IR Manual.

References:
(a) 29 CFR 1910.120
(b) 29 CFR 1926.65
(c) Navy/Marine Corps Installation Restoration Manual (IR Manual)

1. **Names of key personnel and health and safety personnel.** 29 CFR 1910.120(b)(2) and 29 CFR 1926.65(b)(2)	**YES**	**NO**
a. Are key personnel identified in the HASP? Comment:		
b. Are health and safety personnel, including alternates, identified in the HASP? Comment:		
2. **Has a site-specific safety & health risk analysis been accomplished for each site task and operation found in the workplan?** 29 CFR 1910.120(b)(4)(ii)(A) and 29 CFR 1926.65(b)(4)(ii)(A)		
a. Does the HASP address methods to deal with potential safety problems on the site? Comment:		
b. Has an adequate risk analysis for each site task and operation been provided? Comment:		
c. Does the risk analysis include as a minimum: Chemical contaminants Affected media Concentrations Potential routes of exposure Associated health effects Comment:		
d. Are appropriate levels of PPE identified for each site task and operation? Comment:		
3. **Employee Training** 29 CFR 1910.120(b)(4)(ii)(B) and 29 CFR 1926.65(b)(4)(ii)(B) (note this paragraph refers to specific requirements found in 29 CFR 1910.120(e) and 29 CFR 1926.65(e)).		
a. Does the HASP indicate that all on-site employees meet appropriate training requirements? Regulatory Reference: Certificates are to be provided per 29 CFR 1910.120(e)(6), 29 CFR 1910.65(e)(6), and IR Manual para. 11.13. Comment:		
b. Have all on-site employees received initial 40-hour training and certificates either provided or provisions for the certificates being provided on-site? Regulatory Reference: 29 CFR 1910.120(e)(3), 29 CFR 1926.65(e)(3), and IR Manual para. 11.6.1 Comment:		
c. Do all supervisory personnel have 8-hour supervisory training and are certificates either provided or provisions for the certificates being provided on-site? Regulatory Reference: 29 CFR 1910.120(e)(4), 29 CFR 1926.65(e)(4), and IR Manual para. 11.6.1 Comment:	**YES**	**NO**

d. Do all employees working on-site have a minimum of three days actual field experience under the direction of a skilled supervisor? Regulatory Reference: 29 CFR 1910.120(e)(3)(i), 29 CFR 1926.65(e)(3)(i), and IR Manual para. 11.6.1 Comment:		
e. Is refresher training current and are certificates provided or provisions made for the certificates to be provided on-site? Regulatory Reference: 29 CFR 1910.120(e)(8), 29 CFR 1926.65(e)(8), and IR Manual para. 11.6.1 Comment:		
f. Have employees been trained to recognize the symptoms and signs of over-exposure to chemical hazards? Regulatory Reference: 29 CFR 1910.120(c)(8) and 29 CFR 1926.65(c)(8) Comment:		
g. Have employees been trained in First Aid/CPR as necessary? Regulatory Reference: 29 CFR 1926.50(c) Comment:		
h. Have any chemical/physical/toxicological properties of each substance been identified and communicated to the employee? Regulatory Reference: 29 CFR 1910.120(c)(8) and 29 CFR 1926.65.(c)(8) Comment:		
4. **Personnel Protective Equipment** 29 CFR 1910.120(b)(4)(ii)(C) and 29 CFR 1926.65(b)(4)(ii)(C) - refers to more specific requirements found in 29 CFR 1910.120(g)(5) and 29 CFR 1926.65(g)(5). Also, IR Manual para. 12.8 A written program must address the following:		
a. Has the PPE been selected based upon the site hazards? Regulatory Reference: 29 CFR 1910.120(g)(5)(i), 29 CFR 1926.65(g)(5)(i), and IR Manual para. 12.8.1 Comment:		
b. Has the use and limitations of the PPE been described? Regulatory Reference: 29 CFR 1910.120(g)(5)(ii), 29 CFR 1926.65(g)(5)(ii), and IR Manual para. 12.8.3 Comment:		
c. Has the work mission duration been described? Regulatory Reference: 29 CFR 1910.120(g)(5)(iii), 29 CFR 1926.65(g)(5)(iii), and IR Manual para. 12.8.3 Comment:		
d. Have decontamination and disposal procedures been established? Regulatory Reference: 29 CFR 1910.120(g)(5)(iv), 29 CFR 1926.65(g)(5)(v), and IR Manual para. 12.8.3 Comment:		
e. Have employees been properly fitted with the PPE and trained in its use? Regulatory Reference: 29 CFR 1910.120(g)(5)(vi), 29 CFR 1926.65(g)(5)(vi), and IR Manual paras. 11.6.2 and 12.8.3 Comment:		
f. Have employees been trained in proper donning and doffing procedures? Regulatory Reference: 29 CFR 1910.120(g)(5)(vii), 29 CFR 1926.65(g)(5)(vii), and IR Manual para. 12.8.3 Comment:		
g. Have inspection procedures been established? Regulatory Reference: 29 CFR 1910.120(g)(5)(viii), 29 CFR 1926.65(g)(5)(viii), and IR Manual para. 12.8.3 Comment:		
h. Are procedures established to monitor the **effectiveness** of the PPE program? Regulatory Reference: 29 CFR 1910.120(g)(5)(ix), 29 CFR 1926.65(g)(5)(ix), and IR Manual para. 12.8.3 Comment:		
i. Are provisions for limitations of use of the PPE in temperature extremes and for heat stress described? Are other appropriate medical considerations included? Regulatory Reference: 29 CFR 1910.120(g)(5)(x), 29 CFR 1926.65(g)(5)(x), and IR Manual para. 12.8.3 Comment:		
5. **Medical Surveillance** 29 CFR 1910.120(b)(4)(ii)(D) and 29 CFR 1926.65(b)(4)(ii)(D) - this refers to specific requirements found in 29 CFR 19190.120(f) and 29 CFR 1926.65(f). Also, IR Manual para. 12.6 The HASP must include site specific medical monitoring provisions. This should include respirator clearance exams as	**YES**	**NO**

well as other specific tests specified by the examining physician after he/she reviews the site specific information.		
a. Has site specific medical surveillance requirements been included in the HASP? Has all necessary information been provided to the physician? Regulatory Reference: 29 CFR 1910.120(f)(6), 29 CFR 1926.65(f)(6), and IR Manual para. 12.6.5 Comment:		
b. Was the examination performed by or under the supervision of a certified occupational medicine physician? Regulatory Reference: IR Manual para. 12.6.4 Comments:		
6. **Air Monitoring** 29 CFR 1910.120(b)(4)(ii)(E) and 29 CFR 1926.65(b)(4)(ii)(E) - refers to more specific comments found in 29 CFR 1910.120.(h) and 29 CFR 1926.65(h), also IR Manual para. 12.7		
a. Does the HASP include the frequency and types of air monitoring? Regulatory Reference: 29 CFR 1910.120(b)(4)(ii)(E), 29 CFR 1910.120(h)(3), 29 CFR 1926.65(b)(4)(ii)(E) and IR Manual para. 12.7 Comment:		
b. Does the HASP describe methods for personal monitoring? Regulatory Reference: 29 CFR 1910.120.(b)(4)(ii)(E) and 29 CFR 1926.65(b)(4)(ii)(E) Comment:		
c. Does the HASP describe environmental sampling? Regulatory Reference: 29 CFR 1910.120.(b)(4)(ii)(E) and 29 CFR 1926.65(b(4)(ii)(E) Comment:		
d. Are the various types of instrumentation for site sampling described as well as methods for maintenance and calibration? Regulatory Reference: 29 CFR 1910.120.(b)(4)(ii)(E) and 29 CFR 1926.65(b)(4)(ii)(E) Comment:		
7. **Site Control** 29 CFR 1910.120(b)(4)(ii)(F) and 29 CFR 1926.65(b)(4)(ii)(F) - refers to specific requirements found in 29 CFR 1910.120(d) and 29 CFR 1926.65(d) The following items must be contained in the site control section of the HASP:		
a. Is a site description and map provided to include size, location, etc.? Regulatory Reference: 29 CFR 1910.120(d)(3) and 29 CFR 1926.65(d)(3) Comment:		
b. Have site work zones been established? Regulatory Reference: 29 CFR 1910.120(d)(3) and 29 CFR 1926.65(d)(3) Comment:		
c. Is the use of the "buddy system" established? Regulatory Reference: 29 CFR 1910.120(d)(3) and 29 CFR 1926.65(d)(3) Comment:		
d. Have type(s) of site communications, including alerting means for emergencies, been described? Regulatory Reference: 29 CFR 1910.120(d)(3) and 29 CFR 1926.65(d)(3) Comment:		
e. Are safe operating procedures or safe work practices described? Regulatory Reference: 29 CFR 1910.120(d)(3) and 29 CFR 1926.65(d)(3). See IR Manual para. 12.5 for specific SOPs Comment:	**YES**	**NO**
f. Has the nearest medical assistance source been described, civilian <u>and</u> military (as appropriate)? Regulatory Reference: 29 CFR 1910.120(d)(3) and 29 CFR 1926.65(d)(3) Comment:		

8. **Emergency Response Plan** 29 CFR 1910.120(b)(4)(ii)(H) and 29 CFR 1926.65(b)(4)(ii)(H) refers to specific requirements found in 1910.120(l) and 1926.65(1) The plan should provide sufficient detail to ensure prompt, safe mitigation of potential site emergencies. The plan should indicate how emergencies would be handled at the site and how the risks associated with a response would be minimized. Specific Department of the Navy arrangements should be explicitly stated; in particular, identify any involvement with the Navy Medical Department. **Emergency telephone numbers listed in the plan should be verified by the reviewer.**	**YES**	**NO**
a. Has pre-emergency planning been completed? State/Local/Local Emer Planning Committee NOSC/NOSCDR Hazardous Materials Team Medical Treatment Facility Ambulance Navy Medical Department Medical Treatment Facility Ambulance Regional Poison Control Center Agency for Toxic Substances & Disease Registry Other Regulatory Reference: 29 CFR 1910.120(l)(2)(i), 29 CFR 1926.65(1)(2)(i), and IR Manual paras. 12.9.2, 12.9.3, 12.9.5 Comment:		
b. Have personnel roles, lines of authority and communications been established? Regulatory Reference: 29 CFR 1910.120(l)(2)(ii), 29 CFR 1926.65(1)(2)(ii) and IR Manual para. 12.9.2 Comment:		
c. Is emergency recognition and prevention discussed? Regulatory Reference: 29 CFR 1910.120(l)(2)(iii), 29 CFR 1926.65(1)(2)(iii) and IR Manual para. 12.9.2 Comment:		
d. Have safe distances and places of refuge been described by specific maps and written descriptions for each site? Regulatory Reference: 29 CFR 1910.120(l)(2)(iv), 29 CFR 1926.65(1)(2)(iv) and IR Manual para. 12.9.2 Comment:		
e. Have site security and control measures been described? Regulatory Reference: 29 CFR 1910.120(l)(2)(v), 29 CFR 1926.65(1)(2)(v), and IR Manual para. 12.9.2 Comment:		
f. Have evacuation routes and procedures been described by specific maps and written descriptions for each site? Does this include the route to the MTF? Regulatory Reference: 29 CFR 1910.120(l)(2)(vi), 29 CFR 1926.65(1)(2)(vi), and IR Manual paras. 12.9.2 and 12.9.5 Comment:		
g. Are decontamination measures, not discussed elsewhere in the HASP, described? Is the priority for field decontamination vice emergent medical assistance discussed? Regulatory Reference: 29 CFR 1910.120(l)(2)(vii), 29 CFR 1926.65(1)(2)(vii) and IR Manual para. 12.9.2 Comment:		
h. Have provisions for emergency medical treatment and first aid been established? Who is providing the assistance? Civilian MTF? Civilian Ambulance? Navy MTF? Navy Ambulance? Are these facilities equipped and trained? Regulatory Reference: 29 CFR 1910.120(l)(2)(viii), 29 CFR 1926.65(1)(2)(viii), and IR Manual paras. 12.9.2, 12.9.3 and 12.9.5 Comment:		
i. Has information on the chemical hazard(s) been provided to the MTF/ambulance personnel? Regulatory Reference: IR Manual paras. 12.9.2, 12.9.3, 12.9.5 Comment:		
j. Have emergency alerting and response procedures been established? Regulatory Reference: 29 CFR 1910.120(l)(2)(ix), 29 CFR 1926.65(1)(2)(ix), and IR Manual para. 12.9.2 and 12.9.5		

Comment:		
k. Are the telephone numbers listed for emergency response correct? Regulatory Reference: 29 CFR 1910.120(l)(2)(ix), 29 CFR 1926.65(1)(2)(ix), and IR Manual paras. 12.9.3 and 12.9.5 Comment:		
l. Are the site topography, layout, and prevailing weather conditions described? Regulatory Reference: 29 CFR 1910.120(l)(3)(i)(A), 29 CFR 1926.65(1)(3)(i)(A), and IR Manual para. 12.9.3 Comment:		
m. Are PPE and emergency equipment provided and their location clearly indicated? Regulatory Reference: 29 CFR 1910.120(l)(2)(xi), 29 CFR 1926.65(1)(2)(xi), and IR Manual paras. 12.9.2 and 12.9.5 Comment:		
n. Are procedures to report incidents to Local, State, Navy, and other authorities listed? Regulatory Reference: 29 CFR 1910.120(l)(3)(i)(B), 29 CFR 1926.65(1)(3)(i)(B), and IR Manual para. 12.9.3 Comment:		
o. Are procedures to rehearse the plan included? Regulatory Reference: 29 CFR 1910.120(l)(3)(iv), 29 CFR 1926.65(1)(3)(iv), and IR Manual para. 12.9.3 Comment:		
p. Are procedures to review and update the plan included? Regulatory Reference: 29 CFR 1910.120(l)(3)(v), 29 CFR 1926.65(1)(3)(v), and IR Manual para. 12.9.3 Comment:		
q. Are procedures to evaluate and critique emergency response and follow-up included? Regulatory Reference: 29 CFR 1910.120(l)(2)(x), 29 CFR 1926.65(1)(2)(x), and IR Manual para. 12.9.3 Comment		
9. **Confined Space Entry Procedures** 29 CFR 1910.120(b)(4)(ii)(I), 29 CFR 1926.65(b)(4)(ii)(I), and IR Manual para. 12.3.l If these are required, they must be in accordance with 29 CFR 1910.120(j)(9), 29 CFR 1910.146 and 29 CFR 1926.65(j)(9)		
10. **Spill Containment Program** 29 CFR 1910.120(b)(4)(ii)(J) and 29 CFR 1926.65(b)(4)(ii)(J) refers to specific requirements in 29 CFR 1910.120(j), 29 CFR 1926.65(j) and IR Manual para. 12.3.m Elements to be potentially addressed include: Drum and container handling Opening of drums Material handling equipment Radioactive wastes Shock sensitive wastes Laboratory waste packs Sampling drum and container contents Shipping and transport of drums and containers Appropriate procedures for tank and vault entry Does the HASP contain a section discussing site specific spill containment procedures? Comment:	**YES**	**NO**
11. **Decontamination Procedures** 29 CFR 1910.120(k), 29 CFR 1926.65(k), and IR Manual para. 12.8.3 Decontamination procedures should be chosen based on site specific contaminants.		
a. Does the HASP contain site specific decontamination methods for personnel and for equipment? Regulatory Reference: 29 CFR 1910.120(k)(2)(i), 29 CFR 1910.120(k)(2)(ii), 29 CFR 1926.65(k)(2)(i), and 29 CFR 1926..65(k)(2)(ii) Comment:		
b. Are the decontamination methods appropriate for the site conditions and contaminants? Regulatory Reference: 29 CFR 1910.120(k)(i) and 29 CFR 1926.65(k)(i) Comment:		
c. Are decontamination methods monitored by the site safety and health supervisor to determine their effectiveness? Regulatory Reference: 29 CFR 1910.120(k)(iv) and 29 CFR 1926.65(k)(iv) Comment:		

12. Bloodborne Pathogens (29 CFR 1910.1030)		
a. Is there a Bloodborne Pathogens Program? (29 CFR 1910.1030(e))		

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APPENDIX D

OCCUPATIONAL SAFETY AND HEALTH SUBJECT REFERENCE LIST

Biohazards Control
OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations.

29 CFR 1910.1030 Bloodborne pathogens

Bloodborne Pathogens
29 CFR 1910.1030 Bloodborne pathogens

OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations

Chemical Hazards
NIOSH Pocket Guide to Chemical Hazards, NIOSH, 1994, Publication 94-116

Cold Stress
Fundamentals of Industrial Hygiene, National Safety Council, 1996, 4th Edition

NEHC-TM92-6, June 1992, Prevention and Treatment of Heat and Cold Stress Injuries

1995-1996 Threshold Limit Values for Chemical Substances and Physical Agents, and Biological Exposure Indices. American Conference of Governmental Industrial Hygienists, 1995

The Industrial Environment -- Its Evaluation and Control, US Public Health Service, Centers for Disease Control, NIOSH; 1973

Confined Space Entry
29 CFR 1910.146 Permit-required confined spaces

Criteria Document -- Working in Confined Spaces, NIOSH, 1980, Publication 80-106

NAVSEA S6470-AA-SAF-010, Maritime-Shore

NAVSEA S9086-AA-STM-030, Volume 3, Chapter 79, Naval Ships Technical Manual, 1994

Cutting and Welding
29 CFR 1910.252 General requirements
Explosion and Fire
29 CFR 1910.156 Fire brigades

29 CFR 1910.157 Portable fire extinguishers

NFPA Regulations -- National Fire Codes, National Fire Protection Association

Fibers
NEHC-TM91-1 October 1990, Manmade Vitreous Fibers

NEHC-TM91-6 September 1991, Advanced Composite Materials

Hazard Communication - Chemical Hazards
OPNAVINST 4110.2 - Hazardous Material Control and Management (HMC&M), 1989

OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations

29 CFR 1910.1200 Hazard communication

Health and Safety Guidelines
Health and Safety Audit Guidelines - SARA Title 1 Section 126, EPA, 1989, Publication EPA/540/G-89/010

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, 1985, Publication 85-115

Standard Operating Safety Guides, EPA, 1992, Publication 9285.1-03

Heat Stress
Criteria Document -- Occupational Exposure to Hot Environments, NIOSH, 1986, Publication 86-113

Criteria Document -- Working in Hot Environments, NIOSH, 1986, Publication 86-112

Fundamentals of Industrial Hygiene, National Safety Council, 1996, 4th Edition

NEHC-TM92-6, June 1992, Prevention and Treatment of Heat and Cold Stress Injuries

1995-1996 Threshold Limit Values for Chemical Substances and Physical Agents, and Biological Exposure Indices American Conference of Governmental Industrial Hygienists, 1995

The Industrial Environment -- Its Evaluation and Control, US Public Health Service, Centers for Disease Control, NIOSH; 1973

Heavy Equipment
Application Guide for Neutral Grounding in Electric Utility Systems - Part 2 - Grounding of Synchronous Generator Systems Surge Protective Devices, ANSI/IEEE, 1989, C62.92

Guide for Application of Neutral Grounding in Electric Utility Systems - Part 1 - Introduction, ANSI/IEEE, 1987,C62.92

Industrial Hygiene - Technical
NEHC-TM91-2, Revised March 1993, Industrial Hygiene Field Operations Manual

NEHC-TM91-3, Revised May 1993, Industrial Hygiene Sampling Guide for Consolidated Industrial Hygiene Laboratories

Laboratory Safety
OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations

29 CFR 1910.1450, Occupational exposure to hazardous chemicals in laboratories

Medical Concerns
NEHC-TM90-1 March 1990, Occupational Medicine Field Operation Manual

NEHC-TM90-3 August 1990, Emergency Medical Treatment Protocols for Hazardous Materials

NEHC-TM91-5 September 1991, Medical Surveillance Procedures Manual and Medical Matrix (Edition 4)

Noise Hazards
Fundamentals of Industrial Hygiene, National Safety Council, 1996, 4th Edition

OPNAVINST 5100.19 Series -- Navy Occupational Safety and Health (NAVOSH) Program Manual for Forces Afloat, Department of the Navy, Office of the Chief of Naval Operations

OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations

1995-1996 Threshold Limit Values for Chemical Substances and Physical Agents, and Biological Exposure Indices, American Conference of Governmental Industrial Hygienists, 1995

The Industrial Environment -- Its Evaluation and Control, US Public Health Service, Centers for Disease Control, NIOSH; 1973

29 CFR 1910.95, Occupational noise exposure

PCBs
NEHC-TM90-2 May 1990, Polychlorinated Biphenyls (PCBs), Polychlorinated Dibenzofurans (PCDFs), and Polychlorinated Dioxins (PCDDs)

Radiation Hazards
NAVMED P-5055 -- Radiation Health Protection Manual, BUMED, 1992

29 CFR 1910.96, Ionizing radiation

29 CFR 1910.97, Nonionizing radiation

Reproductive Hazards
NEHC-TM92-2 May 1992, Reproductive Hazards in the Workplace: A Guide for Occupational Health Professionals

Respiratory Protection
ANSI Z88.2 -- Practices for Respiratory Protection, 1992

ANSI Z88.5 -- Practices for Respiratory Protection for the Fire Service, 1981

NEHC-TB-1, October 1988, Acceptable Respirators for Use with Ambient Air Breathing Apparatus (AABA)

OPNAVINST 5100.19 Series -- Navy Occupational Safety and Health (NAVOSH) Program Manual for Forces Afloat, Department of the Navy, Office of the Chief of Naval Operations

OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations

29 CFR 1910.134, Respiratory protection

Safe Boat Operations
Federal Requirements for Recreational Boats, DOT, USCG

Flotation Devices, Underwriters Lab, UL 1123/1175

OPNAVINST 5100.19 Series -- Navy Occupational Safety and Health (NAVOSH) Program Manual for Forces Afloat, Department of the Navy, Office of the Chief of Naval Operations

Scaffolding
29 CFR 1910.28, Safety requirements for scaffolding

29 CFR 1926 Subpart L, Scaffolding

Selection, Care, Cleaning of PPE
29 CFR 1910.133, Eye and face protection

Guidelines for the Selection of Chemical Protective Clothing - Performance, Availability, and Sources of Chemical Protective Clothing, DOE, 1991, DE-02357T UCRL-ID-109 106

OPNAVINST 5100.23 Series -- Navy Occupational Safety and Health (NAVOSH) Program, Department of the Navy, Office of the Chief of Naval Operations

Ultraviolet Radiation
Fundamentals of Industrial Hygiene, National Safety Council, 1996, 4th Edition

1995-1996 Threshold Limit Values for Chemical Substances and Physical Agents, and Biological Exposure Indices American Conference of Governmental Industrial Hygienists, 1995

The Industrial Environment -- Its Evaluation and Control, US Public Health Service, Centers for Disease Control, NIOSH; 1973

NEHC-TM92-5 April 1992, Ultraviolet Radiation Guide

Unexploded Ordnance
Ammunition and Explosives Ashore Safety Regulations for Handling, Storing, Production, Renovation and Shipping, NAVSEA OP 5, 1990, Sixth Revision and Change 3, 1995
Ammunition Afloat, NAVSEA OP 4, 1972, Change 18, 1995

OPNAVINST 8023.2C - U.S. Navy Explosives Safety Policies, Requirements, and Procedures, Department of the Navy Explosives Safety Policy Manual, 1986

Utilities
American National Standards, American National Standards Institute

29 CFR 1910.147, The control of hazardous energy (lockout/tagout)

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APPENDIX E

SAMPLER/SAMPLING RECOMMENDATIONS AND STRATEGIES

Table E-1 Recommended Samplers for Various Types of Waste*

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 hydroflouric acid and concentrated alkali solutions
	c) PTFE	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

Waste Type	Recommended Sampler	Limitations
		viscous liquids
	b) Bacon Bomb sampler	Volume restriction 1 L maximum
	c) Kemmerer sampler	May need extra weight

Source: *Sample Collection of Water, Soil, Air and Waste Materials: Protocol, Theory, and Field Applications*

*** These are only recommendations. Consult the applicable regulations to confirm sampler selection recommendations.**

Table E-2 General Sampling Strategies*

Sample Source	Matrix	Sampling Strategy
Drum	Liquid or solid	Collect at least one sample from each drum through bung on top. If contents are stratified collect a sample from each strata.
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 strata.
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.
Waste piles	Solid	Collect one sample per 100 yd^3 of material. Divide pile into a grid and collect sample at the nodes, obtaining a core of 1-2 feet in length at each node.
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 strata 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. If body of water is shallow, ato site or at a point of discharge. Sample should be collected at mid-depth in the channel. Additional samples may be required if multiple discharges and additional streams are present, or for delineation of contamination.
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.

Sample Source	Matrix	Sampling Strategy
		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.

Source: *Sample Collection of Water, Soil, Air and Waste Materials: Protocol, Theory, and Field Applications*

*** These are only recommendations. Consult the applicable regulations to confirm strategy recommendations.**

NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

APPENDIX F

DOT SHIPPING REQUIREMENTS

NOTE:
The information provided in this appendix comes primarily from the Federal regulations (primarily from Titles 40 and 49 of the Code of Federal Regulations). In general, states can impose more stringent requirements than those imposed by the Federal government. Therefore, knowledge and compliance with state and local requirements is imperative.

F.1 EXCLUSIONS FROM EPA HAZARDOUS WASTE MANIFEST REQUIREMENTS FOR SHIPMENTS OF SAMPLES TO AN ANALYTICAL LABORATORY (40 CFR 261.4(D)).

The shipment is exempt from EPA regulations, including the requirement for the use of an EPA Hazardous Waste Manifest, if:

(i) The sample is being transported to a laboratory for the purpose of testing
(ii) The sample is being transported back to the sample collector after testing
(iii) The sample is being stored by the sample collector before transport to a laboratory for testing
(iv) The sample is being stored in a laboratory after testing but before it is returned to the sample collector
(v) The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary

In order to qualify for the exemptions listed in (i) and (ii) above, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

(i) Comply with U.S. Department of Transportation, U.S. Postal Service, or any other applicable shipping requirements
(ii) Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

(A) Assure that the following information accompanies the sample:

- The sampling personnel's name, mailing address, and telephone number
- The laboratory's name, mailing address, and telephone number
- The quantity of the sample
- The date of shipment
- A description of the sample

(B) Package the sample so that it does not leak, spill, or vaporize from its packaging. Ensure that absorbent material is used in packaging to contain liquid in the event of leakage or breakage of the sample container.

F.2 EXEMPTIONS FROM EPA HAZARDOUS WASTE MANIFEST REQUIREMENTS FOR SHIPMENTS OF SAMPLES FOR TREATABILITY TESTING B 40 CFR 261.4(E).

The shipment is exempt from EPA regulations, including the requirement for the use of an EPA Hazardous Waste Manifest, if:

(i) The sample is being collected and prepared for transportation by the generator or sample collector; or
(ii) The sample is being accumulated or stored by the generator or sample collector prior to transportation to a laboratory or testing facility; or
(iii) The sample is being transported to the laboratory or testing facility for the purpose of conducting a treatability study.

These exemptions apply to samples that are sampled and shipped for treatability testing provided that:

(i) The quantity of samples used to evaluate each process for each waste stream shall not exceed:
  - 1,000 kg of any non-acute hazardous waste
  - 1 kg of acute hazardous waste
  - 2500 kg of soils, water, or debris contaminated with acute hazardous waste
(ii) The mass of each sample shipment does not exceed:
  - 1,000 kg of any non-acute hazardous waste
  - 1 kg of acute hazardous waste

kg of soils, water, or debris contaminated with acute hazardous waste

NOTE:
An acute hazardous waste is defined in 40 CFR 261.33 as:

- EPA Hazardous Wastes numbers F020, F021, F022, F023, F026, F027 (wastes from the production and processing of chlorophenols or tetra-, penta-, or hexachlorobenzenes)
- Commercial chemical products, manufacturing chemical intermediates or off-specification commercial chemical products or manufacturing chemical intermediates
- any residue or contaminated soil, water or other debris resulting from the cleanup of a spill of those chemicals listed in 40 CFR 261.33(e) and (f)

Included on these lists are the commonly encountered chemicals chlorobenzene, dichlorobenzene, 1,2,4,5-tetrachlorobenzene, 2-butanone (methylethylketone), DDT, methylene chloride, trichlorophenol, pentachlorophenol, trichloroethylene, tetrachloroethane, and tetrachloroethylene.

(iii) The sample must be packaged so that it will not leak, spill, or vaporize from its packaging during shipment and the following requirements are met:

(A) Comply with U.S. Department of Transportation, U.S. Postal Service, or any other applicable shipping requirements

(B) Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

- Assure that the following information accompanies the sample:
  - The name, mailing address, and telephone number of the sample originator
  - The laboratory's name, mailing address, and telephone number
  - The quantity of the sample
  - The date of shipment
  - A description of the sample, including its EPA Hazardous Waste Number

(iv) The laboratory to which the sample is shipped has an appropriate RCRA permit or interim status <u>or</u> is exempt from the regulations under 40 CFR 261.4(f). This exemption requires that the laboratory has an EPA Identification Number, has notified the Regional Administrator or State Director (if located in an authorized state) at least 45 days before conducting treatability studies, and complies with the rest of the paragraphs of э261.4(f) which specify operating, record keeping, reporting and disposal requirements.

(v) The generator or sampling personnel maintains the following records for a period ending 3 years after completion of the treatability study:

(A) Copies of the shipping documents.

(B) A copy of the contract with the facility conducting the treatability study.

(C) Documentation showing:

- The amount of waste shipped under this exemption
- The name, address, and EPA identification number of the laboratory or testing facility that received the waste
- The date the shipment was made
- Whether or not unused samples and residues were returned to the generator

(vi) The generator reports the information required in (v)(C) above in its biennial report.

F.3 SHIPPING CONTAINER MARKING AND LABELING.

F.3.1 DOT Hazardous Materials. All packages that contain DOT Hazardous Materials must be labeled as specified by 49 CFR 172.400 and subsequent Parts. This labeling requirement applies both to the sample containers that contain hazardous materials and to the shipping container in which the sample containers are packed. The following labeling is required:

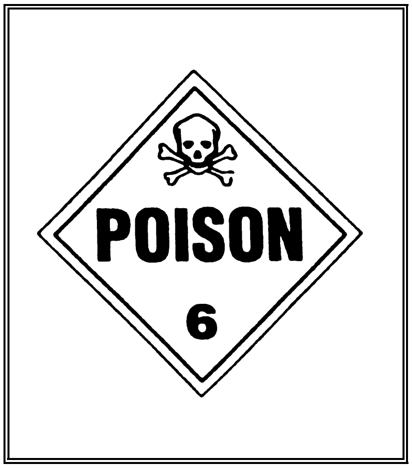
- Proper shipping name and identification number from э172.101 Table, Column 4. (Do not include the word "Waste" in the description unless an EPA Hazardous Waste Manifest is used for the shipment.)
- Technical name in parenthesis in association with the proper shipping name
- DOT shipping label, at least 3.9 inches on a side. **Figure F-1** is a partial list of the labels specified by DOT. Contact shipping company or DOT for instructions for labels of Hazardous Materials
- PCB label, 6" square, if any of the samples contain over 50 ppm PCBs. Small PCB labels (11/16" x 1" or 1" x 2") are available for application to sample containers.

If the contents of a shipping container have different hazard classes, the shipping container must be labeled as required for each class of hazardous material contained therein.

Class 3, Guide 26: Flammable

Background color: Red

Class 6, Guide 55: Poison

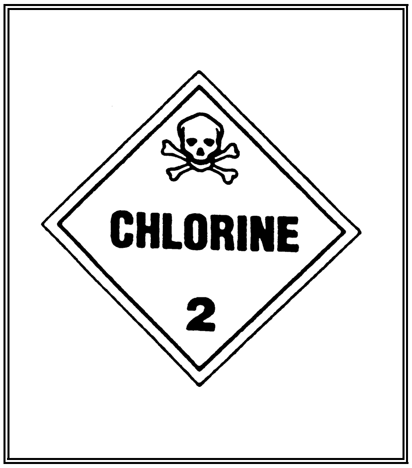


Background color: White

Class 8, Guide 59: Corrosive

Color: Black on white background

Class 2, Guide 15: Chlorine



Background color: White

Figure F-1 DOT Hazardous Material Labels

The following information should appear on the shipping container:

- Shipping Container Number, as shown on the **Chain-of-Custody** Record
- Name and address of shipper
- Ship to address
- "Chain-of-Custody Record enclosed"

Figure F-2 describes the proper container labeling for DOT Hazardous materials where an EPA Hazardous Waste Manifest is required.

F.3.2 Environmental (Non-Hazardous) Samples. Hazardous material labels may not be used on containers that do not contain hazardous materials. Only the following information should appear on the shipping container:

- Container Number
- Name, address and phone number of shipper
- Ship to address and phone number
- "Chain-of-Custody Record enclosed"

If the courier requires that a description of the contents be on the container, use "Non-hazardous Environmental Samples For Chemical Analysis".

Each shipping container may be sealed with a chain of custody seal.

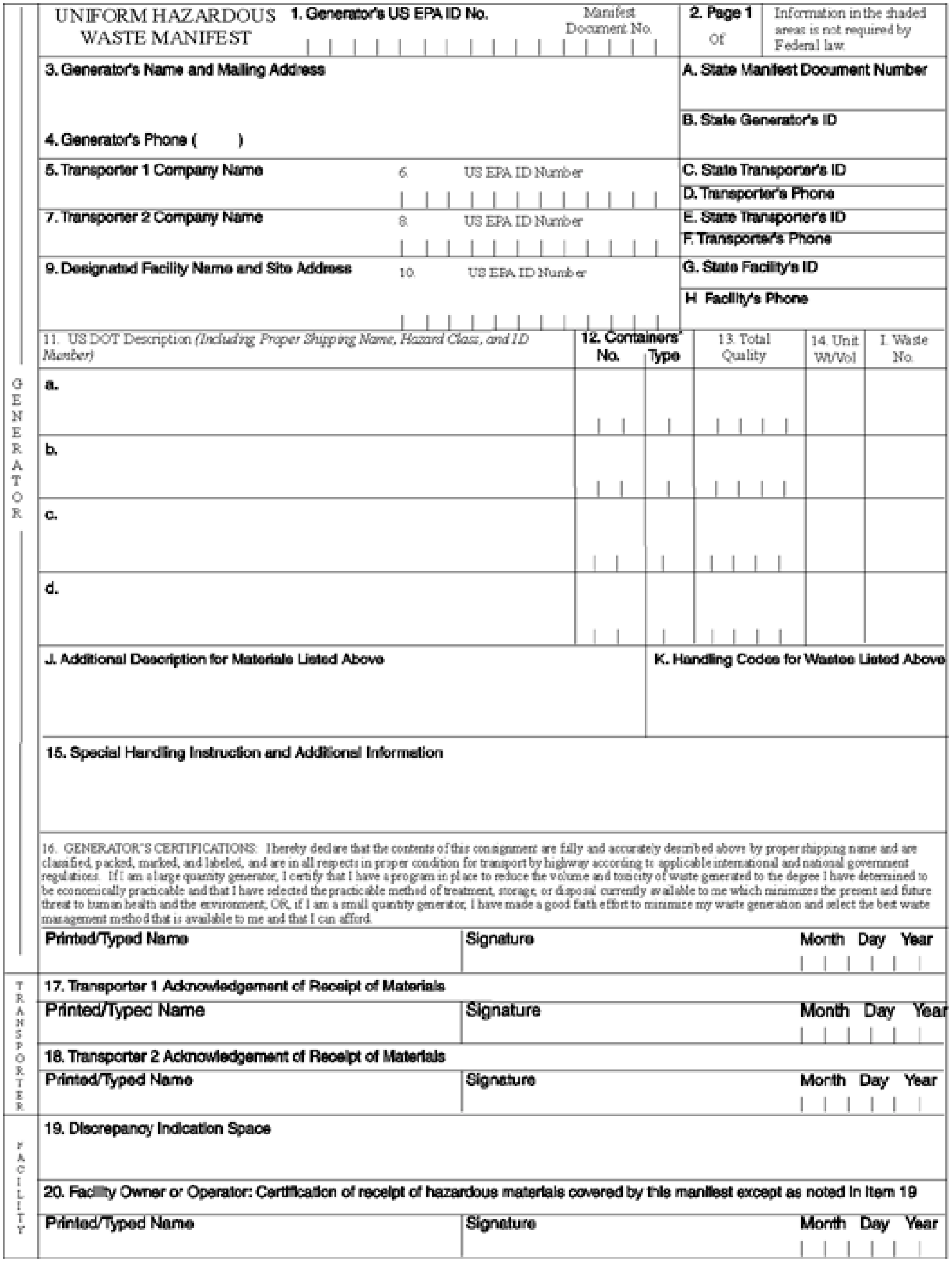
F.4 DOT SHIPPING NAMES - HAZARDOUS MATERIALS.

F.4.1 Determination Of Proper Shipping Name. The sample description must be exactly as written in 49 CFR 172.101 Table. Abbreviations may not be used unless specifically authorized or required by the DOT regulations.

- The sample description of a hazardous material must be identified by the entry of an "X" placed before the proper shipping name in the column captioned "HM". The "X" may be replaced by "RQ", if appropriate (i.e., Class 9).
- The description for material offered for transportation as "limited quantity" must include the words "Limited Quantity" or "Ltd Qty" following the basic description.
- The shipping paper may contain additional information concerning the material provided the information is not inconsistent with the required description.

The shipping description of hazardous material must include the following information on the shipping paper shown in the following sequence:

- The proper shipping name ('172.101 Table, Column 2)
- The hazard class or division prescribed for the material ('172.101 Table, Column 3)
- The identification number prescribed for the material ('172.101 Table, Column 4)
- The packing group, if any, prescribed for the material ('172.101 Table, Column 5)
- Technical and chemical group names may be entered in parentheses between the proper shipping name and hazard class or following the basic description. An appropriate modifier, such as "contain" or "containing" may be used.
- *"n.o.s." and other generic descriptions except Class 9:* the technical name of the hazardous material must be entered in parentheses in association with the basic description.
- *Mixtures:* If a hazardous material is a mixture or solution of two or more hazardous materials, the technical names of at least two components most predominately contributing to the hazards of the mixture or solution must be entered on the shipping paper.
- *Hazardous Substances (Class 9):* If the proper shipping name for a material that is a hazardous substance does not identify the hazardous substance by name, one of the following descriptions shall be entered, in parentheses, in association with the basic description:
  - The name of the hazardous substance as shown in ϶172.101 Appendix
  - For waste streams (F or K #s), the waste stream number
  - For wastes which exhibit an EPA characteristic or ignitability, corrosivity, reactivity, or Toxicity, the letters "EPA" followed by the word "ignitability", or "corrosivity" or "reactivity" or "Toxicity" as appropriate or the corresponding "D" number as appropriate
  - The letters "RQ" shall be entered on the shipping paper either before or after the basic description
- *Poisonous Materials:* Notwithstanding the hazard class to which a material is assigned, if a liquid or solid material in a package meets the definition of a poison according to DOT regulations, and the fact that it is a poison is not disclosed in the shipping name or class entry, the word "Poison"



UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No.

Manifest Document No.

2. Page 1 of

Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address

A. State Manifest Document Number

B. State Generator's ID

4. Generator's Phone (   )

5. Transporter 1 Company Name

6. US EPA ID Number

C. State Transporter's ID

D. Transporter's Phone

7. Transporter 2 Company Name

8. US EPA ID Number

E. State Transporter's ID

F. Transporter's Phone

9. Designated Facility Name and Site Address

10. US EPA ID Number

G. State Facility's ID

H Facility's Phone

11. US DOT Description *(Including Proper Shipping Name, Hazard Class, and ID Number)*	12. Containers No.	12. Containers Type	13. Total Quality	14. Unit Wt/Vol	I. Waste No.
a.					
b.					
c.					
d.					

J. Additional Description for Materials Listed Above

K. Handling Codes for Wastes Listed Above

15. Special Handling Instruction and Additional Information

16. GENERATOR'S CERTIFICATIONS: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations. If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

Printed/Typed Name | Signature | Month Day Year

GENERATOR

17. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | Month Day Year

18. Transporter 2 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | Month Day Year

TRANSPORTER

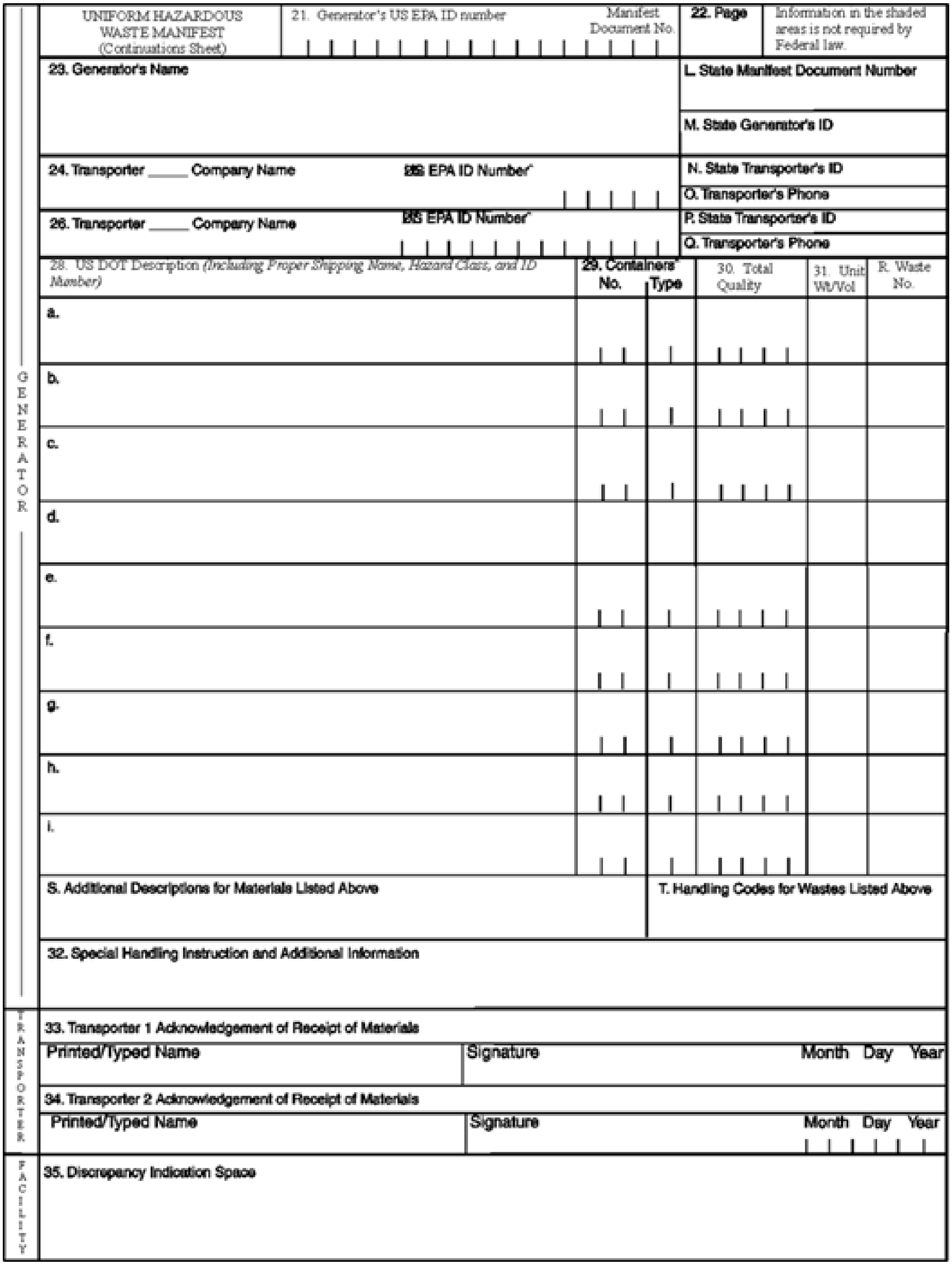
19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19

Printed/Typed Name | Signature | Month Day Year

FACILITY

Figure F-2 DOT Hazardous Material Shipping Container Labeling Where EPA Hazardous Waste Manifest is Required



UNIFORM HAZARDOUS WASTE MANIFEST (Continuations Sheet)	21. Generator's US EPA ID number Manifest Document No.	**22. Page**	Information in the shaded areas is not required by Federal law.

23. Generator's Name

L. State Manifest Document Number

M. State Generator's ID

24. Transporter _____ Company Name

25 EPA ID Number

N. State Transporter's ID

O. Transporter's Phone

26. Transporter _____ Company Name

25 EPA ID Number

P. State Transporter's ID

Q. Transporter's Phone

28. US DOT Description *(Including Proper Shipping Name, Hazard Class, and ID Number)*	**29. Containers No.**	**Type**	30. Total Quality	31. Unit Wt/Vol	R. Waste No.
a.					
b.					
c.					
d.					
e.					
f.					
g.					
h.					
i.					

S. Additional Descriptions for Materials Listed Above

T. Handling Codes for Wastes Listed Above

32. Special Handling Instruction and Additional Information

GENERATOR

33. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name	Signature	Month	Day	Year

34. Transporter 2 Acknowledgement of Receipt of Materials

Printed/Typed Name	Signature	Month	Day	Year

TRANSPORTER

35. Discrepancy Indication Space

FACILITY

Figure F-2 (Cont.) DOT Hazardous Material Shipping Container Labeling Where Hazardous Waste Manifest is Required shall be entered on the shipping paper in association with the shipping description

- ° If the technical name of the compound or principal constituent that causes a material to meet the definition of a poison is not included in the proper shipping name for the material, the technical name shall be entered in parentheses in association with the basic description on the shipping paper
- ° For Division 2.3 materials and Division 6.1, Packing Group I materials which are poisonous by inhalation, the words "Poison-Inhalation Hazard' [and the words "Hazard Zone A", "Hazard Zone B", "Hazard Zone C", or "Hazard Zone D", as appropriate], shall be entered on the shipping paper in association with the shipping description. The word "Poison" need not be repeated if it otherwise appears in the shipping description.

- The total quantity (weight, volume, etc.) of the hazardous material covered by the description (may appear before or after the description).

Examples:

X, Flammable liquids, n.o.s. (contains Xylene and Benzene), 3, UN1993, PG II.

Environmentally hazardous substance, solid, n.o.s., 9, UN3077, PG III, RQ (Adipic Acid)

X, Corrosive liquid, n.o.s. (Caprylyl chloride), 8, UN1760, PG II

X, Flammable liquid, corrosive, n.o.s., 3, Flammable Liquid, UN2924, PG II, (contains Methanol, Potassium hydroxide)

Samples of soil containing PCBs > 20 ppm and TCLP characteristic waste for lead where at least one sample container contains more than one pound of soil (the CERCLA RQ).

NOTE:
An EPA Hazardous Waste Manifest is <u>not</u> required if the sample is being sent for analysis or treatability testing.

RQ, Environmentally hazardous substances, solid, n.o.s., Ltd Qty, 9, UN3077, PG III (POLYCHLORINATED BIPHENYLS [PCBs], D008)

Soil containing PCBs > 20 ppm and TCLP characteristic waste for lead being sent for treatability testing where the total amount of soil being shipped exceeds 1,000 kg.

NOTE:
An EPA Hazardous Waste Manifest <u>is</u> required.

RQ, Hazardous Waste, solid, n.o.s., 9, NA3077, PG III (POLYCHLORINATED BIPHENYLS [PCBs], D008)

Samples of soil containing PCBs > 20 ppm and TCLP characteristic waste for lead where all the sample containers contain less than one pound of soil (the CERCLA RQ).

NOTE:
An EPA Hazardous Waste Manifest is <u>not</u> required if the samples are being sent for analysis or treatability testing. *The shipping container is <u>not</u> classified as hazardous under DOT regulations.*

When a hazardous material and a non-hazardous material are described on the same shipping paper, the hazardous material description entries must be entered first or must be written in a color that clearly contrasts with any description on the shipping paper of non-hazardous materials.

F.4.2 <u>Precedence Of Hazard - Materials Classified In Two Hazard Classes 3, 4, 5, 6 and 8</u>.

1. Determine your <u>DOT hazard class</u> as a <u>"Material"</u>, in order of priority shown in **Table F-1** (as established by 49 CFR 173.2a).

Remember some DOT Hazards are defined by characteristic (e.g., Flash point < 60.5° C (141° F): Flammable liquid (Class 3)). Others are defined by listing (e.g., Lithium batteries and Asbestos are listed as Class 9).

Aniline, for example, is listed with a "+" in column 1 of э172.101 Table. Therefore, it must be shipped as Poison (Class 6.1) regardless of toxicity data.

2. Note all possible names in 49 CFR 172.101 for your sample, per the following priority:

1 <u>Technical names</u> (See Dictionaries and Indexes)
e.g., "dimethyl ketone" is "acetone"

Table F-1 Precedence of DOT Hazard Classes

Priority Ranking	Class/Division	Description
see э173.2(c)	1	Explosive
	5.2	Organic Peroxide
	6.2	Infectious substance
	4.1	Wetted Explosive
1	7	Radioactive
2	2.3	Poisonous gases
3	2.1	Flammable gases
4	2.2	Non-flammable gases
5	6.1 Packing Group I	Poisonous liquids
6	4.2	Pyrophoric material
7	4.1 material	Self-reactive
8 (see following table)	3	Flammable liquids
	8	Corrosive materials
	4.1	Flammable solids
	4.2	Spontaneously combustible materials
	4.3	Dangerous when wet materials
	5.1	Oxidizers
	6.1	Poisonous liquids, Packing Group II, III
9		Combustible liquids
10	Class 9	CERCLA Reportable Quantities

2 <u>Chemical Generic (family) names;</u> e.g., pentyl alcohol is an "Alcohols, n.o.s."

3 <u>End Use</u> of Material; e.g., "Paint"

4 <u>"n.o.s."</u> End Use of Material; e.g., "Dyes, liquid, n.o.s."

5 <u>DOT Class of Hazard</u>; e.g., "Flammable liquids, n.o.s."

If found, note the proper shipping name, hazard class and division, labels required, packaging group, packaging references, etc., as may be applicable.

NOTE:
Remember, if you find your material on DOT's List э172.101, that does <u>not</u> mean it is definitely regulated by the EPA under RCRA.

CAUTION

Be certain your unknown in fact possesses the same hazard class, division, packing group, and subsidiary hazard(s) as listed in э172.101 under the DOT name you chose.

3. See if your unknown or any component of your unknown is listed as a "Hazardous Substance" in э172.101 Appendix A, "List of Hazardous Substances and Reportable Quantities".

   To be considered a Hazardous Substance, it must equal or exceed its reportable quantity (RQ) in one container, and if the listed hazardous substance is only a component of your unknown, it must equal or exceed the corresponding concentration per **Table F-2**:

   NOTE:

   Hazardous substance shipments require additional communications, including the letters "RQ" and, in some cases, the technical name of the hazardous substance (See Step #11).

4. Is the sample a "Solid Waste" as defined under RCRA? Refer to 40 CFR Section 261.2 and state equivalent regulations.

5. See if your sample is, or contains, a "listed waste" in 40 CFR Part 261, Subpart D (Sections 261.31 and 261.32 if it is a process waste, and Sections 261.33(e) and 261.33(f) if it is a chemical product). If found, note the EPA/State Waste designation, the EPA Hazardous Waste Identification Number, and the list where found.

6. Does your sample possess any of the characteristics defined in 40 CFR Part 261, Subpart C? If so, then it is a "Characteristics Hazardous Waste" (EPA) (See 40 CFR Sections 261.20 through 261.24 and state equivalents thereto.)

7. If a proper shipping name was provided on э172.101 Table per #2 above, then that also will be the proper shipping name for the waste ... you must insert the word "waste" before the DOT proper shipping name if, and only if, the waste is required to be manifested by the USEPA. (Reference 49 CFR 172.101(c)(9).

8. If the sample was not given a proper name as per #2 above but it is considered a hazardous waste by the USEPA vis-à-vis steps 4, 5, and 6 above, then the proper shipping name and hazard class is:

   - "Hazardous waste, liquid or solid, n.o.s."
   - Hazard Class 9

9. If the sample is not specifically named on э172.101 Table, and the material has more than one hazard, refer to the priority of hazard lists, 49 CFR 173.2a and name according to the highest priority hazard. Remember, in some cases, samples having more than one hazard may require multiple labeling, so refer also to 49 CFR 172.402. The same is true for mixtures having more than one hazard. Additional descriptions on paper work may be required (49 CFR 172.203).

10. If your sample has a "generic" proper shipping name listed at 49 CFR 172.203(k)(3), {See Below}, you must insert the technical name of the material in parentheses between the proper shipping name and the hazard class. If the sample is a mixture of two or more hazardous materials, the description must include the technical names of at least two components "most predominantly contributing to the hazards of the mixture". Refer to 49 CFR 172.203(k).

11. Note that it is required that a recognizable technical (chemical) name be included in the proper shipping name for all poisons. If your sample is a poison and has a non-technical proper shipping name such as an "end use" or "n.o.s." designation, then a technical name will also have to be inserted after the proper shipping name

Table F-2 Concentration Limits for DOT Hazardous Substances

	Concentration by Weight	
RQ Pounds	Percent	ppm
5,000	10	100,000
1,000	2	20,000
100	0.2	2,000
10	0.02	200
1	0.002	20

given by the table (and before the hazard class). Poisons by inhalation also require the "Hazard Zone" to be specified on shipping papers. Refer to 49 CFR 172.203(m).

12. If your sample is a "Hazardous Substance" listed in the э172.101 Appendix, don't forget to note the reportable quantity (or "RQ") and to proceed appropriately. See 49 CFR 172.203(c)(2) and other communications and reporting requirements for "Reportable Quantities" (e.g. Markings: 49 CFR 172.302.6 and state equivalents).

13. Empties: Any empty container that had previously contained a DOT hazardous material is to be considered still a hazardous material for shipping purposes. Unless it is "sufficiently cleaned of residue and purged of vapors to remove any potential hazard" it must meet all DOT requirements except as specified at э173.29.

    An empty is regulated by the EPA as a "Hazardous Waste" if it previously contained either a hazardous waste or a product as referenced in 40 CFR Section 261.33(c). A container that contained anything listed in Section 261.33(e) or (f) must be managed as described in Section 261.33(c). The definition of when a container is "empty" (i.e., no longer regulated) under EPA rules can be found at 40 CFR Section 261.7(b).

    In any case, it is allowed, and it is recommended practice, to insert the phrase "RESIDUE: Last Contained" at the beginning of the proper shipping name used to ship and transport hazardous empties. (See 49 CFR 172.203(e)).

14. Be sure to check all other aspects and details as may affect the proper communication of hazards, as may be found in 49 CFR Part 172, all Subparts.

15. If all else fails, call:
    EPA HOTLINE (800) 424-9346

F.4.3 Improper Shipping Names. The following is a list of some of the more common mistakes being made in identifying and naming hazardous materials for shipment.

1. Names for U. S. surface shipments must be taken from the table at 49 CFR 172.101.

In most instances, ICAO (IATA) rules may be used for international or intranational shipment by air [49 CFR 171.11]; and the IMDG Code may be used if all or part of the transportation is by vessel [49 CFR 171.12]; and Canadian TDG rules may be used for shipments originating in Canada [49 CFR 171.12a].

Secondary publications such as the DOT Emergency Response Guide should **NEVER** be used as a reference for selecting proper shipping descriptions. Such publications may contain a mixture of international and intranational descriptions and may be considerably out of date.

2. Never ship a hazardous material using a name that is not listed with the hazard class actually exhibited by the material shipped (See 49 CFR 173.2a for multiple hazard materials). The only materials for which you need not determine the actual hazard prior to selecting a proper shipping description are materials listed in э172.101 with a "+" in column 1 [49 CFR 172.101(b) (1)]. This notation fixes the proper shipping name and hazard class, regardless of the hazard presented. Also, specific materials listed as "Class 9", and **which present no higher hazard**, are always Class 9 [49 CFR 173.140].

3. Never identify a mixture of two or more hazardous materials by the name of one of them, whether or not the word "mixture" or "solution" is added. The proper shipping name must always identify all hazardous constituents. Often, a generic or "n.o.s." proper shipping name must be used for such mixtures. The words "mixture" or "solution" are used to identify mixtures of a hazardous material with other non-hazardous materials [49 CFR 172.101(c)(10)].

NOTE:

"Solution" identifies an homogeneous liquid mixture which will not separate during transportation. "Mixture" means any other mixture. [49 CFR 171.8.]

4. The word "Waste" or "Hazardous Waste" in a DOT proper shipping name indicates that the material is required to be shipped on a hazardous waste manifest by the USEPA. Wastes which are not required to be shipped on a hazardous waste manifest by Federal rules (e.g., state-regulated "hazardous waste", Asbestos waste, samples exempted from manifesting requirements, etc.)

may not be called "Waste" [49 CFR 171.101(c)(9).

5. It may never be assumed that a material may be shipped out under the same description by which it was received. Many situations may make that description incorrect. For example:

   - The original shipment may have been made under an exemption to which you must become a party
   - Changes in packaging or quantity per package may alter the requirements or exclusions applicable to the material
   - The material may have somehow been changed (by contamination, deterioration, etc.) and no longer exhibit the same hazard or no longer meet the same description
   - The material may now be a "waste" as regularly defined, and be subject to additional requirements, or
   - The original shipping description may simply have been incorrect

6. Never simply assume that a material poses the worst possible hazard and ship it under a "protective" proper shipping name. This is a bad practice for at least three reasons:

   a. It is **bad public relations** to tell all who observe (e.g., neighbors, agencies, employees, etc.) that you are shipping large quantities of extraordinarily hazardous materials when you are, in fact, not.
   b. It may be **dangerous**. Emergency responders to an incident involving such shipments may take unnecessary or incorrect precautions, exacerbating the incident.
   c. Last, but not least, it is **illegal** to ship non-hazardous materials under a DOT hazard class (e.g., 49 CFR 172.101(c)(13).

7. "Hazardous substances" in packages less than their Reportable Quantity (RQ) are not hazardous substances by US DOT definition (49 CFR 171.8). If they do not exhibit any other DOT hazard, they are not DOT hazardous materials and may not be shipped as such. If they do exhibit another DOT hazard, they should be shipped under that hazard but do not require "RQ" communications.

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

APPENDIX G

KEY NAVY AND EPA POINTS OF CONTACT, NAVY ENVIRONMENTAL LABORATORIES, AND ENVIRONMENTAL HOTLINES

G.1 NAVAL POINTS OF CONTACT. Listed below are contacts and sources of information for specific program areas and technical elements. It is important to be aware of Environmental Protection Agency (EPA), state and local regulatory agency information sources as well as those of the Navy which may be helpful to field sampling and testing personnel.

G.1.1. Chief of Naval Operations.

Chief of Naval Operations
Director, Environmental Protection, Safety and Occupational Health Division, N45
Crystal Plaza 5
2211 South Clark Place
Arlington, VA 22244-5108
703-602-1738 DSN 332-1738

Chief of Naval Operations
Shore Facilities Engineering Division
Environmental Planning Branch, N44E
Crystal Plaza 5
2211 South Clark
Arlington, VA 22244-5108
(703) 325-0032 DSN 221-0032

G.1.2 Naval Sea Systems Command (NAVSEASYSCOM).

G.1.2.1 NAVSEA Headquarters.

Commander
Naval Sea Systems Command
Office of Environmental Protection/ Occupational Safety & Health (SEA 00T)
2531 Jefferson Davis Hwy
Arlington, VA 22242-5160
703-602-3594, DSN 332-3594

Commander
Naval Sea Systems Command
Office of Counsel (SEA 00L)
2531 Jefferson Davis Hwy
Arlington, VA 22242-5160

Commander
Naval Sea Systems Command
Naval Shipyard BRAC Implementation Group (SEA 074)
2531 Jefferson Davis Hwy
Arlington, VA 22242-5160

G.1.2.2 NAVSEA DET Radiological Affairs Support Office (RASO).

Officer in Charge
Naval Sea Systems Command Detachment, Radiological Affairs Support Office
Naval Weapons Station
P.O. Drawer 260
Yorktown, VA 23691-0260
804-887-4692, DSN 953-4692

G.1.2.3 NAVSEA Multi-Service Environmental Laboratories. (See **Figure G-1**)

Commander
Norfolk Naval Shipyard (Code C134.12)
Environmental Laboratory, B184
Portsmouth, VA 23709
(804) 396-3028

Commander
Portsmouth Naval Shipyard (Code 134)
Materials Testing Laboratory, Bldg 20-2
Portsmouth, NH 03804-5000
(207) 438-1890 or DSN 684-1890

Commander
Puget Sound Naval Shipyard (Code 134)
PSNSY Environmental Laboratory, Bldg 371
Bremerton, WA 98314-5000
(360) 476-8091

Commander
Long Beach Naval Shipyard (Code 1182)
300 Skipjack Road, Building 129
Long Beach, CA 90807
(310) 547-6541 or DSN 360-6541

Commander
Pearl Harbor Naval Shipyard (Code 134)
401 Ave. E, Suite 124
Pearl Harbor, HI 96860
(808) 474-3045

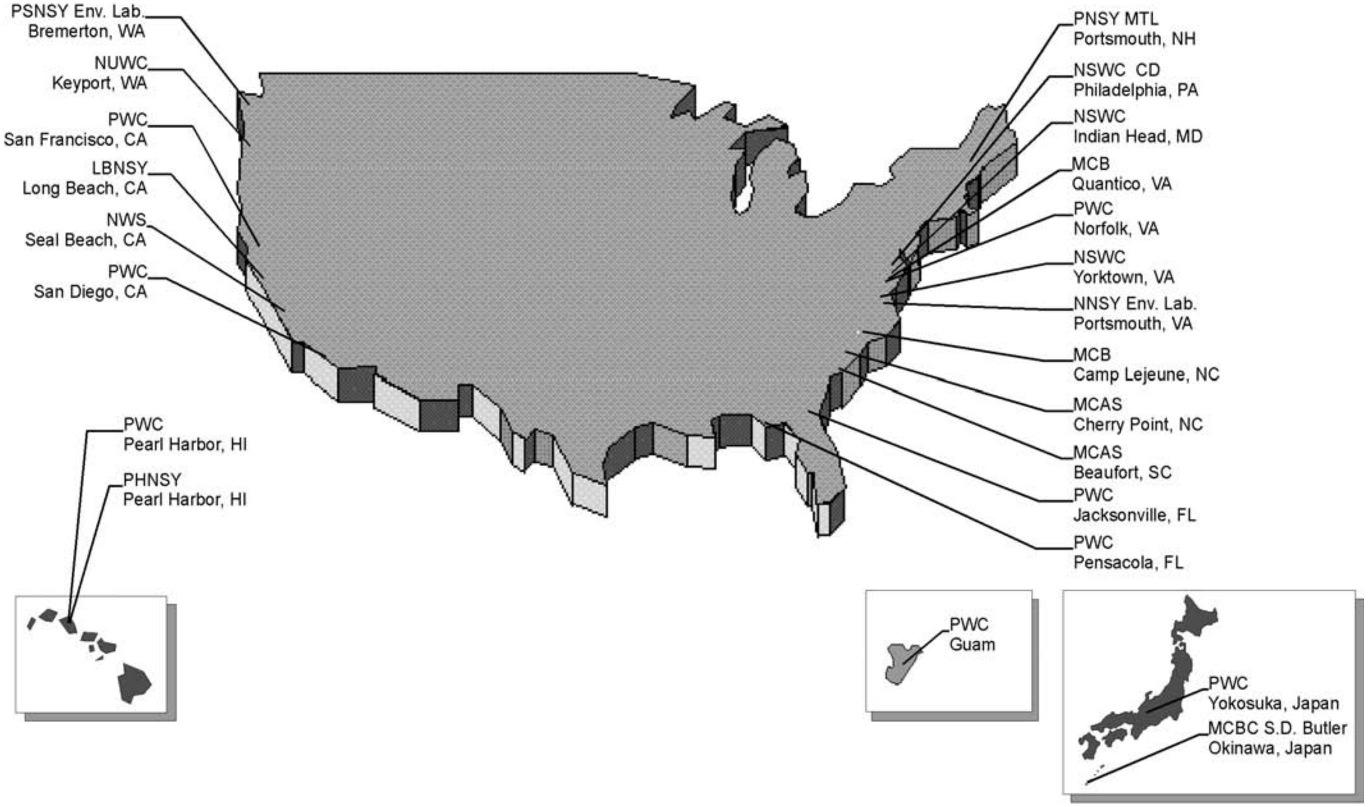

Figure G-1 Map of Multi-Service Environmental Laboratories

Commander
Naval Surface Warfare Center
Indian Head Division
101 Strauss Avenue, Bldg 1864
Indian Head, MD 20640-5035
(301) 743-6521, DSN 354-6521

Commander
Naval Surface Warfare Center
Indian Head Division, Yorktown Detachment
P.O. Drawer 160
Yorktown, VA 23691-0160
(804) 887-4311, DSN 953-4311

Commander
Naval Surface Warfare Center
Carderock Division, SSES
U.S. Naval Base
Philadelphia, PA 19112-5083
(215) 897-7502

Commander
Naval Undersea Warfare Center
Keyport Division, ETSD
610 Dowell Street
Keyport, WA 98345-7610
(360) 396-2501, DSN 396-7683

Commanding Officer
Naval Weapons Station Seal Beach
800 Seal Beach Blvd
Seal Beach, CA 90740-5000
(310) 626-7124

Director
SUPSHIP Portsmouth Environmental Det
Charleston Naval Base, Bldg 30
Charleston, SC 29408-2020

G.1.3 Naval Facilities Engineering Command (NAVFACENGCOM).

Commander
Naval Facilities Engineering Command
(Code 40)
200 Stovall Street
Alexandria, VA 22332-2300
703-325-0295, DSN 221-0295

G.1.3.1 NAVFAC Engineering Field Divisions.

Commander
Atlantic Division, Code 18
Naval Facilities Engineering Command
1510 Gilbert Street
Norfolk, VA 23511-2699
804-322-4800, DSN 262-4800

Commander
Pacific Division, Code 18
Naval Facilities Engineering Command
Pearl Harbor, HI 96780-7300
808-474-3211, DSN 474-4519

Commanding Officer
Northern Division, Code 18
Naval Facilities Engineering Command
10 Industrial Highway, Mail Stop 82
Lester, PA 19113-2090
610-595-0567, DSN 443-0567 Ext. 115

Commanding Officer
Southern Division, Code 18
Naval Facilities Engineering Command
2155 Eagle Drive, P.O. Box 190010
North Charleston, SC 29419-9010
803-743-0600, DSN 563-0600

Commanding Officer
Southwest Division, Code 18
Naval Facilities Engineering Command
1220 Pacific Highway
San Diego, CA 92132-5190
619-532-1396, DSN 522-1396

G.1.3.2 NAVFAC Engineering Field Activities.

Commanding Officer
Engineering Field Activity Chesapeake, Code 18
Naval Facilities Engineering Command
Washington Navy Yard
901 M Street SE
Washington, DC 20374-5018
202-685-3241, DSN 325-3241

Commanding Officer
Engineering Field Activity Mediterranean, Code 18
PSC 810, Box 51
FPO AE 09619-0051
39-81-509-7537, DSN 314-625-3109

Commanding Officer
Engineering Field Activity Midwest, Code 900
2703 Sheridan Road, Suite 120
Great Lakes, IL 60088-5600
(708) 688-4693, DSN 792-4693

Commanding Officer
Engineering Field Activity Northwest, Code 18
Naval Facilities Engineering Command
19917 7th Avenue NE
Poulsbo, WA 98370-7570
306-396-0072, DSN 744-0072

Commanding Officer
Engineering Field Activity West, Code 18
900 Commodore Drive
San Bruno, CA 94066-5006

415-244-2504, DSN 494-2504

G.1.3.3 NAVFAC Engineering Service Center.
Commanding Officer
Naval Facilities Engineering Service Center
Director, Environmental Department, ESC 40
1100 23rd Street
Port Hueneme, CA 93043-4370
805-982-3584, DSN 551-3584

G.1.3.4 Naval Public Works Center Environmental Laboratories. (See **Figure G-1**)
Commanding Officer
Public Works Center, Norfolk (Code 930)
9742 Maryland Avenue
Norfolk, VA 23511
(804) 445-8850

Commanding Officer
Public Works Center, Jacksonville (Code 330)
Naval Air Station, Jacksonville
Bldg. 902, Code 330
Jacksonville, FL 32212
(904) 778-9584

Commanding Officer
Navy Public Works Center, Pensacola (Code 910)
Naval Air Station, Pensacola
310 John Tower Road
NAS Pensacola, FL 32508
(904) 452-4728/3642, DSN 922-4728

Commanding Officer
Public Works Center, San Diego (Code 910)
Environmental Lab
NAS North Island, Bldg M-9
San Diego, CA 92135
(619) 545-8432

Commanding Officer
Navy Public Works Center
San Fransisco Bay Laboratory, Bldg 415
Naval Station, Treasure Island
San Fransisco, CA
(510) 302-5426, DSN 672-5426

Commanding Officer
US Public Works Center, Pearl Harbor (Code 330)
Bldg X-11
Pearl Harbor, HI 96860-5470
(808) 474-3704

Commanding Officer
U.S. Navy Public Works Center, Guam (Code 690)
Naval Activities, Guam
PSC 455, Box 195
FPO AP 96540-2937
(671) 339-3220
(761) 339-2106

Commanding Officer
U.S. Public Works Center, Yokosuka (Code 940)
FPO AP 96349
011-81-311-743-9061, DSN 243-9061

G.1.4 U.S. Marine Corps Multi-Service Environmental Laboratories. (See **Figure G-1**)
Commanding General
Marine Corps Base Camp Lejeune
AC/S EMD/EQAB
PSC Box 20004
Camp Lejeune, NC 28542-0004
(910) 451-5977, DSN 484-5977

Commanding General
Marine Corps Base Camp S.D. Butler
AC/S Facilities, Unit 35001
FPO AP 96373-5001
011-81-611-745-0427, DSN 645-0427

Commanding Officer
MCAS Beaufort
Public Works Department
P.O. Box 55019
Beaufort, SC 29904-5019
(803) 522-6511, DSN 832-6511

Commanding General
Marine Corps Base Quantico
Natural Resource and Environmental Affairs Lab
3040 McCawley Ave.
Quantico, VA 22134-5053
(703) 784-4030, DSN 278-4030

Commanding General
Marine Corps Air Station Cherry Point
Facilities Maintenance Department Laboratory
PSC Box 8006
MCAS Cherry Point, NC 28533-0006
(919) 466-2520, DSN 582-2520

G.1.5 Defense Environmental Security Corporate Information Management (DESCIM) Program Office.
Executive Director
Room 12S49
200 Stovall Street
Alexandria, VA 22332-2300
703-325-0002, DSN 221-0002

G.1.6 Chief of Naval Education and Training.
Chief of Naval Education and Training
Attn: Environmental, Code 441
250 Dallas Street
Pensacola, FL 32508-5200
904-452-4022, DSN 922-4022

G.1.7 Naval School, Civil Engineer Corps Officers (CECOS).

Commanding Officer
Naval School, Civil Engineer Corps Officers
Attn: Code 09E
3502 Goodspeed Street, Suite 1
Port Hueneme, CA 93043-4336
805-982-6529, DSN 551-6529

G.1.8 Naval Occupational Safety and Health and Environmental Training Center.

Commanding Officer
NAVOSHENVTRACEN
9080 Breezy Point Crescent
Norfolk, VA 23511-3998
804-445-8778, DSN 565-8778

G.1.9 Energetic Materials Sampling and Analysis Expertise.

Commander
Explosive Ordnance Disposal Group ONE
3626 Guadalcanal Road
San Diego, CA 92155-5584
(619) 437-0723, DSN 577-0723

Commander
Explosive Ordnance Disposal Group TWO
2520 Midway Road, Suite 100
Norfolk, VA 23521-3323
(804) 464-8452, DSN 680-8452

Commanding Officer
Naval Weapons Station Seal Beach
Test Systems and Science Laboratory
800 Seal Beach Blvd.
Seal Beach, CA 90740
(310) 626-7141

Commanding Officer
Naval Surface Warfare Center
(Codes 30, 40, 50, 56, 90)
Indian Head Division
101 Srauss Avenue

Indian Head, MD 20640-5000
(301) 743-4680

Commanding Officer
Naval Surface Warfare Center
Indian Head Division (Code 930)
Yorktown, VA 23691-0161

Commander
Naval Air Warfare Center
Weapons Division
1 Administration
China Lake, CA 93555-6001

Commanding Officer
Naval Undersea Warfare Center Division
610 Dowell Street
Keyport, WA 98345-0580
(360) 396-2501

G.1.10 Navy Regional Environmental Coordinators. (See **Figure G-2**)

Regions I and II
(Maine, Vermont, New Hampshire, Connecticut, Massachusetts, Rhode Island, New York, New Jersey)
Commander
Submarine Group TWO (N55429)
Naval Submarine Base
New London, Box 1
Groton, CT 063459-5100

Region II
(Caribbean)
Commander (N09003)
Fleet Air Caribbean
PSC 1008
Box 3037
FPO AA 34051-8000

Region III
(Delaware, District of Columbia, Maryland, Pennsylvania, Virginia, West Virginia)
Commander (N61463)
Naval Base Norfolk
1530 Gilbert Street, Suite 200
Norfolk, VA 23511-2797

Region IV
(Alabama, Florida, Georgia, Kentucky, Mississippi, North Carolina, South Carolina, Tennessee)
Commander (N09697)
Naval Base Jacksonville
Box 102
Naval Air Station
Jacksonville, FL 32212-0102

Region V
(Illinois, Indiana, Michigan, Minnesota, Ohio, Wisconsin)
Commander (N00210)
Naval Training Center
Staff Civil Engineer, Bldg. 5
2701 Sheridan Road
Great Lakes, IL 60088-5000
Region VI
(Texas, Louisiana, Oklahoma, Arkansas, New Mexico)
Commander (N66734)
Naval Reserve Force

4400 Dauphine Street
New Orleans, LA 70146-5000

Region VII
(Iowa, Kansas, Missouri, Nebraska)
Commander (N68330)
Naval Reserve Readiness Command Region 13
2701 Sheridan Road
Great Lakes, IL 60088-5026

Region VIII
(Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming)
Commander (N68308)
Naval Reserve Readiness Command Region 20
410 Palm Avenue
San Francisco, CA 94130

Region IX
(Arizona, Southern California, Nevada, Hawaii, American Samoa, Guam)
Commander (N00242)
Naval Base San Diego
937 N. Harbor Drive
San Diego, CA 92132-5100

(Northern California)
Commander (N61447)
Naval Base San Francisco
Treasure Island
410 Palm Drive
San Francisco, CA 94130-0411

(Hawaii, Midway Islands)
Commander (N61449)
Naval Base Pearl Harbor
Box 110
Pearl Harbor, HI 96860-5020

Region X
(Alaska, Washington, Oregon, Idaho)
Commander (N68742)
Naval Base Seattle
SUBASE Bangor
1103 Hunley Road
Silverdale, WA 98315-5000

(Guam)
Commander
U.S. Naval Forces Marianas

PSC 489
FPO AP 96536-0051

(Japan)
Commander (N57006)
U.S. Naval Forces Japan
PSC 473 Box 12
FPO AP 96349-0051

(Korea)
Commander (N62894)
U.S. Naval Forces Korea
Unit 15250
APO AP 96205-0023

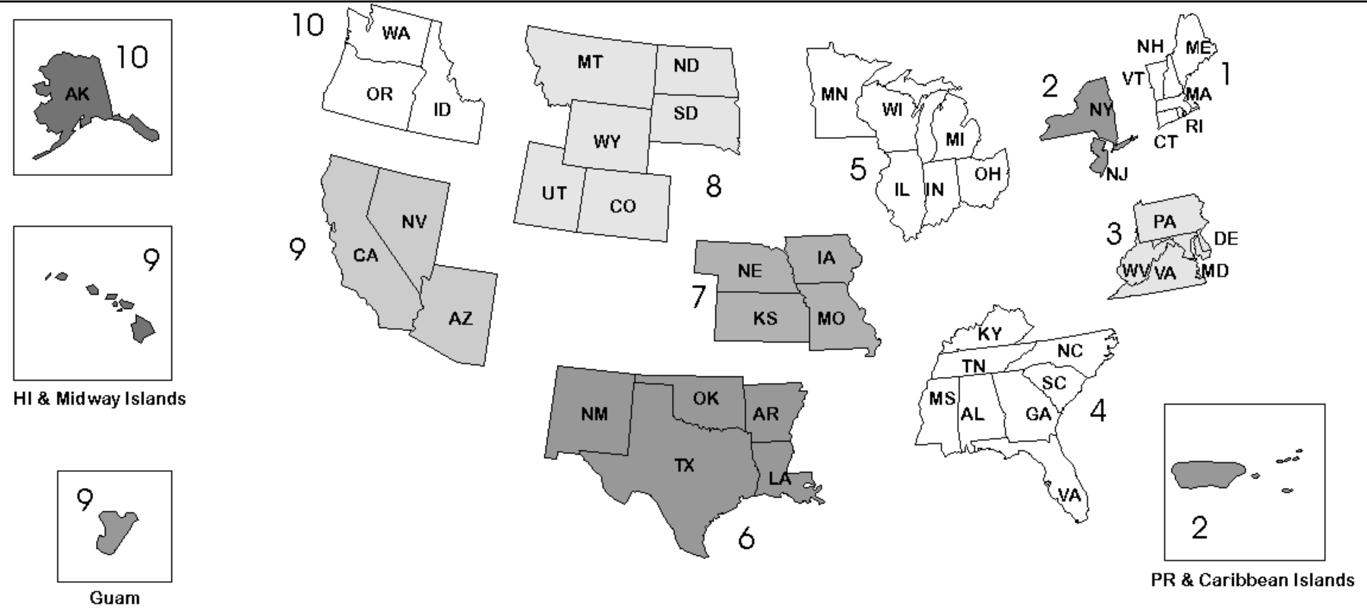

Figure G-2 Navy Area and Regional Environmental Coordinators

CINCPACFLT
AEC for Regions 9 and 10

COMNAVBASE Seattle
Seattle, WA (10)

COMNAVBASE San Francisco
San Francisco, CA (9 Lead)

COMNAVBASE San Diego
San Diego, CA (9)

COMNAVBASE Guam
Guam (9)

COMNAVBASE Pearl
Pearl Harbor, HI (9)

COMNAVRESFOR
AEC for Regions 7 and 8

COMNAVRESFOR
New Orleans, LA (7)

COMNAVRESFOR
New Orleans, LA (8)

CNET
AEC for Regions 5 and 6

NTC Great Lakes
Great Lakes, IL (5)

COMNAVRESFOR
New Orleans, LA (6)

CINCLANTFLT
AEC for Regions 1, 2, 3, and 4

COMSUBGRU TWO
Groton, CT (1 Lead)

COMFAIRCARIB
Puerto Rico (2)

COMNAVBASE Norfolk
Norfolk, VA (3)

COMNAVBASE Jacksonville
Jacksonville, FL (4)

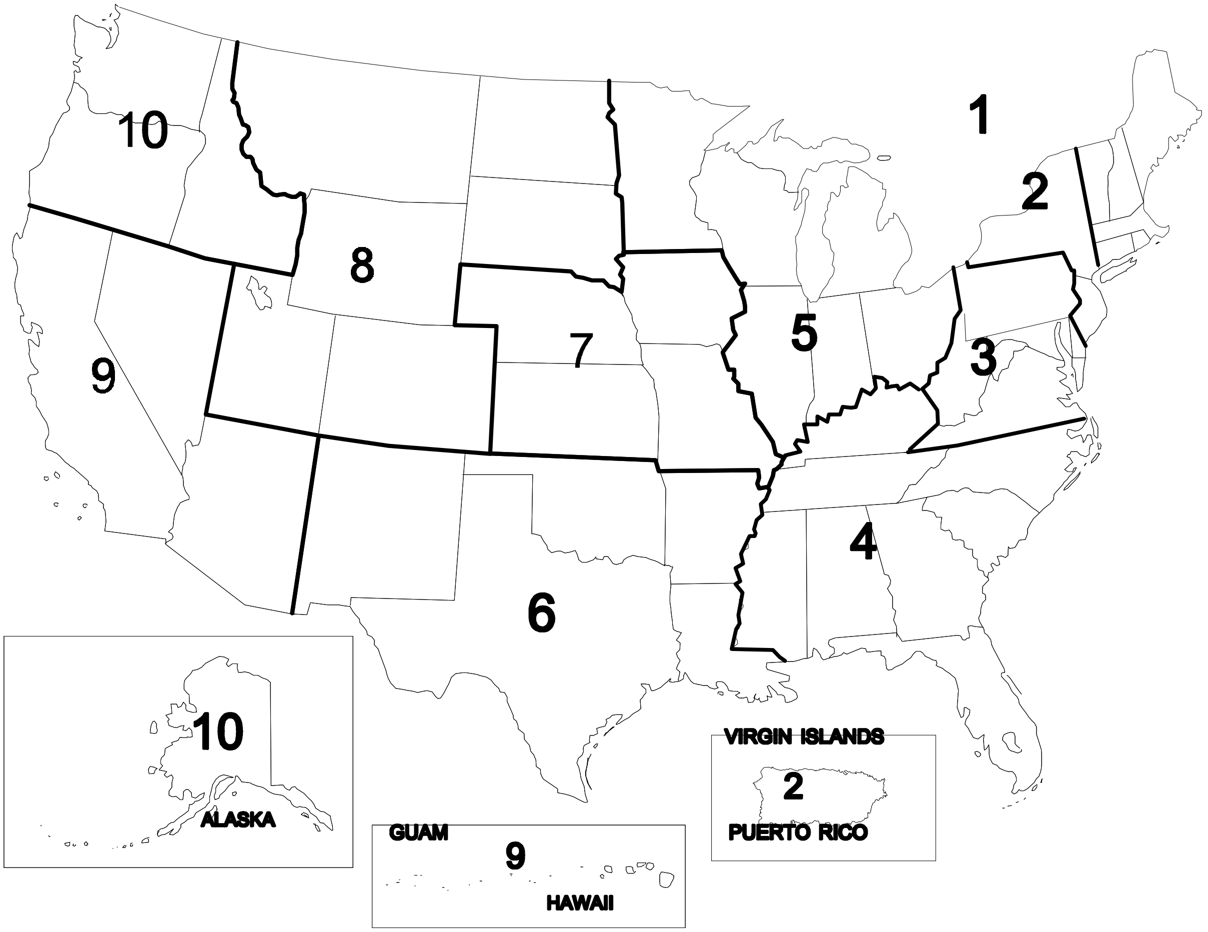

Figure G-3 EPA Regional Offices

G.2 SELECTED EPA HEADQUARTERS ASSISTANT ADMINISTRATORS AND THE EPA REGIONAL OFFICES.

G.2.1 Selected EPA Headquarters Assistant Administrators.

Water   (202) 260-5700
Air and Radiation(202) 260-7400
Solid Waste (202) 260-4610
Pesticides / Toxic Substances   (202) 260-2902
Enforcement (202) 260-4134

G.2.1 EPA Regional Offices. (See **Figure G-3**)

Region I
Environmental Protection Agency
John F. Kennedy Federal Building
One Congress Street
Boston, MA 02203
General Phone: 617-565-3420
Hazardous Waste Ombudsman: 617-565-5758

Region II
Environmental Protection Agency
Jacob K. Javitz Federal Building
26 Federal Plaza
New York, NY 10278
General Phone: 212-264-2657
Hazardous Waste Ombudsman: 212-264-2980

Region III
Environmental Protection Agency
841 Chestnut Building
Philadelphia, PA 19107
General Phone: 215-597-9800
Hazardous Waste Ombudsman: 215-597-6197
General Information Hotline: 215-597-2176
(within region): 800-438-2474

Region IV
Environmental Protection Agency
345 Courtland Street, N.E.
Atlanta, GA 30365
General Phone: 404-347-4727
Hazardous Waste Ombudsman: 404-347-3004

Region V
Environmental Protection Agency

77 West Jackson Boulevard
Chicago, IL 60604-3507
General Phone: 312-353-2000
Hazardous Waste Ombudsman: 312-886-7577
Hotline (within Region): 800-621-8431

Region VI
Environmental Protection Agency
First Interstate Bank Tower at Fountain Place
1445 Ross Avenue 12th Floor Suite 1200
Dallas, TX 75202-2733
General Phone: 214-655-6444
Hazardous Waste Ombudsman: 214-655-8526
Environmental Emergency Hotline
(within Region): 214-655-2222

Region VII
Environmental Protection Agency
726 Minnesota Avenue
Kansas City, KS 66101
General Phone: 913-551-7000
Hazardous Waste Ombudsman: 913-551-7050
Regional Action Line
(within Region): 800-848-4568
Emergency Response: 913-236-3778

Region VIII
Environmental Protection Agency
999 18th Street, Suite 500
Denver, CO 80202-2405
General Phone: 303-293-1603
Hazardous Waste Ombudsman: 303-294-1111
Emergency Response Hotline: 303-294-1788
(within Region): 800-227-8914

Region IX
Environmental Protection Agency
75 Hawthorne Street
San Francisco, CA 94105
General Phone: 415-744-1702
Hazardous Waste Ombudsman: 415-744-2124

Region X
Environmental Protection Agency
1200 Sixth Avenue
Seattle, WA 98101
General Phone: 206-553-4973
Hazardous Waste Ombudsman: 206-553-6901

G.2.2 <u>EPA Dockets</u>.

Resource Conservation and Recovery Act (RCRA)	703-412-9810
Clean Air Act (CAA)	202-260-7548
Underground Storage Tanks (UST)	202-260-9720
Safe Drinking Water Act (SDWA)	202-260-3027
Toxic Substances Control Act (TSCA)	202-260-7099
Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA)	703-305-5805

G23 <u>Environmental Hotlines</u>

Air Risk Information Support Center Hotline	919-541-0888
Army Environmental Information Response Line	800-872-3845
Asbestos Ombudsman Clearinghouse/Hotline	800-368-5888
Chemical Emergency Preparedness Program (CEPP) Hotline	800-535-0202
Chemical Transportation Emergency Center	800-424-9300
Chemical Referral Center (CMA)	800-CMA-8200
EPCRA Hotline	800-535-0202
Federal Aviation Administration Hotline	202-426-4817
FIFRA/General Pesticide Information	800-858-7378
Hazardous Technical Information Services	800-848-4847
Hazardous Waste Ombudsman Program	202-260-9361
Indoor Air Quality Information Clearinghouse	800-438-4318
National Response Center (Report chemical releases, radiological incidents)	800-424-8802
National Safety Council	708-285-1121
National Institute of Occupational Safety & Health (NIOSH)	800-356-4674
National Air Toxics Information Clearinghouse	919-541-0850
Navy CFC and Halon Clearinghouse	703-769-1883
PRO-ACT Helpline	800-233-4356
Pollution Prevention Information Clearinghouse	202-260-1023
RCRA/CERCLA/UST Hotline	800-424-9346
Safe Drinking Water Act (SDWA) Hotline	800-426-4791

Solid Waste Assistance Program	800-677-9424
Stratospheric Ozone Hotline	800-296-1996
Substance Identification	800-848-6538
TSCA Asbestos Hotline	202-554-1404
US DOT Hotline	202-366-4488

NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

APPENDIX H

REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Table H-1 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 H-1** represents the sample handling requirements as they existed at the stated date. 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 FSP.

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 Quality Assurance tests - refer to **Appendix I** 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 Obtaining Contaminant-Free Containers*, April 1990. SDWA sampling containers and cleaning procedures can be found in the *Manual for the Certification of Drinking Water Laboratories*, 1990. The laboratory can furnish containers of the required size and cleanliness.

NOTE:

> Volatile organic analysis (VOA) vials are to have PTFE-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 4°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 Field Sampling Plan.

Filling Instructions. VOA vials with septum caps are to be filled completely with no head space or air pockets. For all other containers, leave adequate head space 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.

Reference. The information provided in the following table lists the requirements for each compliance program from the Code of Federal Regulations (CFR) or from the test method listed in the CFR.

Table H-2 specifies guidance for preserving biological samples, including fish, bottom associated organisms, and algae.

DRINKING WATER (SDWA 40 CFR 141 and 143, as of January 1996) *(Note 1)*

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
Residual chlorine, pH, temperature	N.A. (field measurements)	None		Analyze in field	Some states require certified samplers. Check state requirements.
Volatile Organic Compounds (VOCs) By GC	Four 40-mL glass vial(GC)	Add 1:1 HCl to pH < 2; Cool to 4°C If chlorine is present add 25 mg ascorbic acid prior to HCl addition. Seal bottle and shake vigorously for 1 minute.	Sample vials must be full and free of headspace.	14 days	40 CFR 141 EPA Method 502.2 (GC) rev. 2.0, 1989. For specific volatile compounds review method requirements.
Volatile Organic Compounds (VOCs) By GC/MS	Four 60-mL glass vials (GC/MS)	Add 1:1 HCl to pH < 2; Cool to 4°C If chlorine is present add 25 mg ascorbic acid prior to acid addition. Seal bottle and shake vigorously for 1 minute.	Sample vials must be full and free of headspace.	14 days	40 CFR 141 EPA Method 524.2 (GC/MS) rev 3.0 1989 For specific volatile compounds review method requirements.
Synthetic Organic Chemicals (Pesticides, PCBs)	Two 1-liter amber glass bottles	$HgCl_2$ to 10 mg/L. If chlorine is present add 80 mg sodium thiosulfate. Seal bottle and shake vigorously for 1 minute. Cool to 4°C.	Fill to neck of bottle	7 days until extraction; analysis within 14 days after extraction	40 CFR 141 EPA Method 508 (rev. 3.0 1989)
PCBs (screening)	Two 1-liter amber glass bottles	Cool to 4°C	Fill to neck of bottle	14 days until extraction; analysis within 30 days after extraction	40 CFR 141.24 EPA Method 508A (rev. 1.0 1989)
Disinfection By-Products (Trihalomethanes) and Chlorinated Solvents	Four 40-mL glass vials	Cool to 4°C If chlorine is present add 25 mg ascorbic acid, adjust pH to 4.5 - 5.0 with 0.2 N HCl. Additional information on use of dechlorination and preservation agents must be reviewed in the method.	Sample vials must be full and free of headspace.	14 days	40 CFR 141.24 EPA Method 551 (July, 1990)

DRINKING WATER (Continued)

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
Chlorinated Acids	Two 1-liter amber glass bottles	$HgCl_2$ to 10 mg/L. If chlorine is present add 80 mg sodium thiosulfate. Seal bottle and shake vigorously for 1 minute. Cool to 4°C.	Fill to neck of bottle	14 days until extraction; analysis within 28 days after extraction	40 CFR 141.24 EPA Method 515.1 (rev 4.0 1989)
Primary Metals (Sb, Ba, Be, Cd, Hg, Cr, Ni, Tl, Se)	100-mL polyethylene or glass bottle	$(1+1)HNO_3$ to pH <2 Unpreserved samples may be sent to the laboratory within two weeks then acid preserved, and held 16 hours before analysis.	Fill to neck of bottle	6 months (28 days for mercury)	40 CFR 141 EPA Method 200.7 (rev 4.4 1994) EPA Method 245.1, 245.2, 200.8 Hg
Lead and copper	1-liter polyethylene bottle	$(1+1)HNO_3$ to pH <2 Unpreserved samples may be sent to the laboratory within two weeks, then acid preserved and held 16 hours before analysis.	Fill to neck of bottle	6 months	40 CFR 141.23 EPA Method 200.7 (copper) 200.8, 200.9 (rev 2.2 1994)
Secondary Metals (Al, Fe, Mn, Ag, Zn)	100-mL polyethylene or glass bottle	$(1+1)HNO_3$ to pH <2 Unpreserved samples may be sent to the laboratory within two weeks then acid preserved, and held 16 hours before analysis.	Fill to neck of bottle	6 months	40 CFR 143.4 EPA Method 200.7 (rev 4.4 1994)

DRINKING WATER (Continued)

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
Fluoride	300-mL glass or polyethylene bottle	None	Fill to neck of bottle	1 month	40 CFR 141, Manual Distillation followed by EPA Method 300.0 *(Note 2)*
Silica	50-mL polyethylene bottle	Cool to 4°C	Fill to neck of bottle	7 days	40 CFR 141.23 EPA Method 200.7
Orthophosphate	100-mL polyethylene bottle	Filter on site Cool to 4°C	Fill to neck of bottle	48 hours	40 CFR 141.23 EPA Method 365.1, 300
Calcium	100-mL polyethylene or glass bottle	Conc. HNO_3 to $pH < 2$	Fill to neck of bottle	6 months	40 CFR 141.23 EPA Method 200.7
Chloride	100-mL polyethylene bottle	None	Fill to neck of bottle	28 days	40 CFR 143.4 EPA Method 300.0 (rev 2.1 8/93)
Alkalinity	100-mL glass or polyethylene bottle	Cool to 4°C	Fill to neck of bottle	14 days	Standard Method 2320B
Hardness	100-mL polyethylene or glass bottle	HNO_3 or H_2SO_4 to $pH < 2$	Fill to neck of bottle	6 months	40 CFR 136 EPA Method 130.2, 130.1
Conductivity	100-mL polyethylene bottle	Cool to 4°C	Fill to neck of bottle	28 days	Standard Method 2510B
Turbidity	1-liter polyethylene bottle	Cool to 4°C	Fill to neck of bottle	Not established	40 CFR 141.74 EPA Method 180.1 (rev 2.0 8/93)
Asbestos	100-mL glass or polyethylene bottle	Cool to 4°C	Fill to neck of bottle	Not established	40 CFR 141.23 EPA 100.1 and 100.2
Total Coliform	Sterilized 125-mL polyethylene or glass bottle	0.008% $Na_2S_2O_3$ if residual Cl_2 is present. Cool samples below 10°C.	Fill to neck of bottle	30 hours Surface Water Treatment Rule: 8 hours	40 CFR 141.21, 141.74 Standard Methods 9221A, B, 9222A, B, C, 9221D, 9223 Surface Water Treatment Rule: 9221A, B, C 9222A, B, C 9223
Nitrate	250-mL glass or polyethylene bottle	If chlorinated, cool to 4°C. If not chlorinated, add conc. H_2SO_4 to $pH < 2$.	Fill to neck of bottle	Chlorinated: 28 days Non-chlorinated: 14 days	40 CFR 141.23 EPA Method 300.0 353.2

DRINKING WATER (Continued)					
PARAMETER	**SAMPLE CONTAINERS**	**PRESERVATION**	**FILLING INSTRUCTIONS**	**HOLDING TIME**	**REFERENCE**
Nitrite	50-mL glass or polyethylene bottle	Cool to 4°C	Fill to neck of bottle	48 hours	40 CFR 141.23 EPA Method 300.0 353.2
Radionuclides	1-liter glass or polyethylene bottle	Concentrated HNO_3 to $pH < 2$, except Cesium-134 preserved w/HCl to $pH < 2$. Trititium, Iodine-131 not preserved.	Fill to neck of bottle	Not established	40 CFR 141.25 EPA-600/4-75-008

WASTEWATER NPDES PERMIT SAMPLING (CWA 40 CFR Part 136, as of January 1994) *(Note 1)*

PARAMETER	**SAMPLE CONTAINERS**	**PRESERVATION**	**FILLING INSTRUCTIONS**	**HOLDING TIME**	**REFERENCE**
Dissolved Oxygen, Probe	N.A. - field measurement	None	Glass bottles to top	Within 15 minutes	40 CFR 136.3 Table II
Fecal coliform	Sterilized 125-mL polyethylene or glass bottle	Cool to 4°C; add 0.008% $Na_2S_2O_3$ if residual Cl_2 is present	Fill to neck of bottle	6 hours	40 CFR 136.3 Table II
Biological Oxygen Demand (BOD)	1-liter polyethylene or glass bottle	Cool to 4°C	Fill to neck of bottle	48 hours	40 CFR 136.3 Table II
Chemical Oxygen Demand (COD)	1-liter polyethylene or glass bottle	Add H_2SO_4 to pH < 2; cool to 4°C	Fill to neck of bottle	28 days	40 CFR 136.3; Table II
Total Organic Carbon (TOC)	1-liter glass bottle	Cool to 4°C; add H_2SO_4 or HCl or H_3PO_4 to pH < 2	Fill to neck of bottle	28 days	40 CFR 136.3 Table II
Oil and grease	1-liter glass bottle	Add H_2SO_4 or HCl to pH < 2; Cool to 4°C	Fill to neck of bottle	28 days	40 CFR 136.3 Table II
Metals, Total (For dissolved metals samples must be filtered on site)	100-mL polyethylene or glass bottle	HNO_3 to pH < 2	Fill to neck of bottle	6 months, except mercury 28 days	40 CFR 136.3 Table II
Chromium, hexavalent (Cr^{+6})	200-mL polyethylene bottle	Cool to 4°C	Fill to neck of bottle	24 hours	40 CFR 136.3 Table II
Pesticides	Two 1-liter amber glass bottles	Cool to 4°C Add NaOH or H_2SO_4 to pH 5-9; Add 0.008% $Na_2S_2O_3$ if residual Cl_2 is present. *(Note 3)*	Fill to neck of bottle	7 days until extraction. Analyze within 40 days after extraction.	40 CFR 136.3 Table II
PCBs	Two 1-liter amber glass bottles	Cool to 4°C	Fill to neck of bottle	7 days until extraction. Analyze within 40 days after extraction.	40 CFR 136.3 Table II

GROUNDWATER (RCRA 40 CFR 260, as of April 1995) *(Note 1)*

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
pH, temperature, conductivity	N.A. - field measurements	None		pH - 24 hours	SW-846 Chapter 2, Table 2-33, rev 2, 9/94
Volatile Organics	Four 40-mL glass vials	Cool to 4°C pH < 2 with HCl or H_2SO_4 Presence of chlorine requires proper addition of $Na_2S_2O_3$ prior to adding acid. *(Note 2)*	Sample vials must be full and free of headspace	14 days	SW-846 Chapter 4, Table 4.1, rev 2, 9/94
Semivolatile Organics	Two 1-liter amber glass bottles	Cool to 4°C, keep away from light. If chlorine is present add 3 mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle	7 days until extraction; analysis within 40 days after extraction	SW-846 Chapter 4, Table 4.1, rev 2, 9/94
Pesticides PCBs Herbicides	Two 1-liter amber glass bottles	Cool to 4°C, keep away from light. If chlorine is present add 3 mL 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle	7 days until extraction; analysis within 40 days after extraction	SW-846 Chapter 4, Table 4.1, rev 2, 9/94
Dioxins and Furans	Two 1-liter amber glass bottles	Cool to 4°C, keep away from light. If chlorine is present add 0.008% $Na_2S_2O_3$	Fill to neck of bottle	7 days until extraction; analysis within 40 days after extraction	SW-846 Chapter 2, Table 2-33, rev 2, 9/94
Metals, Dissolved	1-liter polyethylene or glass bottle	Filter through 0.45 µm filter, HNO_3 to pH <2;	Fill to neck of bottle	6 months (28 days for mercury)	SW-846 Chapter 2, Figure 2-4C, rev 2, 9/94
Metals, Total	1-liter polyethylene or glass bottle	HNO_3 to pH < 2	Fill to neck of bottle	6 months (28 days for mercury)	SW-846 Chapter 2, Table 2-33, rev 2, 9/94
Cyanide	1-liter polyethylene or glass bottle	50% NaOH to pH >12, If chlorine is present add 0.06 g ascorbic acid per liter, cool to 4°C, If other oxidizing agents are suspected refer to Method 9010 for preservation.	Fill to neck of bottle	14 days	SW-846 Chapter 2, Table 2-33, rev 2, 9/94
Total Organic Halides (TOX)	1-liter polyethylene or glass bottle	HCl, H_2SO_4 or solid $NaHSO_4$ to pH < 2, Cool to 4°C	Fill to neck of bottle	28 days	SW-846 Chapter 2, Table 2-33, rev 2, 9/94
Radiological Tests	1-liter polyethylene or glass bottle	HNO_3 to pH < 2	Fill to neck of bottle	6 months	SW-846 Chapter 2, Table 2-33, rev 2, 9/94

OTHER WATERS AND NON-AQUEOUS LIQUIDS (RCRA) *(Note 1)*

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
Semivolatile Organics	Two 1-liter amber glass bottles	Cool to 4°C; keep away from light. If chlorine is present, add 3mL of 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle	7 days until extraction; analysis within 40 days after extraction	SW-846 Chapter 4, Table 4.1, rev 2, 9/94
Pesticides PCBs Herbicides	Two 1-liter amber glass bottles	Cool to 4°C; keep away from light. If chlorine is present, add 3mL of 10% $Na_2S_2O_3$ per gallon.	Fill to neck of bottle	7 days until extraction; analysis within 40 days after extraction	SW-846 Chapter 4, Table 4.1, rev 2, 9/94
Volatile Organics	Two 40-mL glass vials	Cool to 4°C; pH < 2 with HCl or H_2SO_4. Presence of chlorine requires proper addition of $Na_2S_2O_3$ prior to adding acid. *(Note 4)*	Sample vials must be full and free of headspace	14 days	SW-846 Chapter 4, Table 4.1, rev 2, 9/94
Metals, Total	1-liter polyethylene or glass bottle	HNO_3 to pH < 2	Fill to neck of bottle	6 months	SW-846, Chapter 2, Table 2-33, rev 2, 9/94
Cyanide	1-liter polyethylene bottle	50% NaOH to pH >12; If chlorine is present, add 0.06g ascorbic acid per liter. Cool to 4°C. If other oxidizing agents are suspected, refer to Method 9010 for preservation.	Fill to neck of bottle	14 days	SW-846, Chapter 2, Table 2-33, rev 2, 9/94
Mercury	1-liter polyethylene or glass bottle	HNO_3 to pH < 2	Fill to neck of bottle	28 days	SW-846 Method 7470A

OTHER WATERS AND NON-AQUEOUS LIQUIDS (CERCLA Low and Medium Concentrations) *(Note 1)*

Refer to Navy IR Program					

OTHER WATERS AND NON-AQUEOUS LIQUIDS (CERCLA High Hazard Protocol) *(Note 1)*

Refer to Navy IR Program					

SOILS, SLUDGES, AND SOLID WASTE (RCRA) *(Note 1)*

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
Volatile Organics	125-mL widemouth glass container sealed with a septum	Cool to 4°C	Fill completely, no headspace	14 days	SW-846, Chapter 4, Table 4.1, rev 2, 9/94
Volatile Organics in concentrated waste samples	125-mL widemouth glass container with Teflon lined lid	Cool to 4°C	Fill completely, no headspace	14 days	SW-846, Chapter 4, Table 4.1, rev 2, 9/94
Semivolatile Organics, Pesticides, PCBs and Herbicides	250-mL widemouth glass container with Teflon lined lid	Cool to 4°C	Fill to neck of bottle	Extract within 14 days; analyze within 40 days after extraction	SW-846, Chapter 4, Table 4.1, rev 2, 9/94
Semivolatile Organics Organochlorine-Pesticides, PCBs and Herbicides in concentrated waste samples	125-mL widemouth glass container with Teflon lined lid	Cool to 4°C	Fill to neck of bottle	Extract within 14 days; analyze within 40 days after extraction	SW-846, Chapter 4, Table 4.1, rev 2, 9/94
Dioxins and Furans, (PCDFs, PCDDs)	250-mL wide mouth glass container with Teflon lined lid	Cool to 4°C	Fill to neck of bottle	Extract within 30 days; analyze within 45 days of sampling	SW-846 Method 8280, 9/86
Total Petroleum Hydrocarbons	250-mL widemouth glass container sealed with Teflon lined lid	Cool to 4°C	Fill completely, no headspace	Per sampling plan and state or local requirements	state or local regulations to be reviewed for sampling and holding time.
BTEX	250-mL widemouth glass container with Teflon lined lid	Cool to 4°C	Fill completely, no headspace	14 days	state or local regulations to be reviewed for sampling and holding time.
Oil and grease (in sludge and sediment)	250-mL widemouth glass container sealed with Teflon lined lid	Add HCl to pH < 2 for samples with aqueous layers, Cool to 4°C	Fill to neck of bottle	28 days	SW-846 Method 9071A, rev 1, 9/94
Metals, Total	250-mL wide mouth glass or polyethylene jar	Cool to 4°C	Fill to neck of bottle	Mercury: 28 days Other metals: 6 months	SW-846 Chapter 2, Table 2-33, rev 2, 9/94
Chromium, hexavalent (Cr^{+6})	500-mL plastic jar	Cool to 4°C	Fill to neck of bottle	48 hours	SW-846 Method 7197, 9/86 for certain domestic/industrial wastes

SOLID WASTE (RCRA Hazardous Waste Classification) *(Note 1)*

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
TCLP Characteristics	Sampling plan based on material to be sampled	Cool to 4°C if no physical change will result	Fill per sampling plan	Organics: 14 days Mercury: 28 days Metals: 180 days	SW-836 Method 1311, July, 1992
VOCs (TCLP extract)	TEDLAR^R bags or gas-tight syringes	Cool to 4°C	Fill per laboratory procedure	14 days	SW-846 Method 1311, July, 1992
Metals; Semivolatile Organics; Pesticides (TCLP extract)	Three 1-liter wide-mouth glass bottles	Cool to 4°C; Preserve extract per analyte method, metals; HNO_3 pH < 2.	Fill to neck of bottle	Organics: 14 days Mercury: 28 days Metals: 180 days	SW-846 Method 1311, July, 1992
Total Organic Halides	250-mL amber wide-mouth glass jar	H_2SO_4 pH < 2; Cool to 4°C	Fill completely no headspace	28 days	SW-846 Method 9022B, rev 2, 9/94
pH, Soil and Waste	250-mL amber wide-mouth glass jar or wide mouth plastic bottle	None required	Fill per sampling plan	Analyze as soon as possible. 24 hours maximum	SW-846 Method 9045C, rev 3, 1/95 and Chapter 2, Table 2-33, rev 2, 9/94
pH, Aqueous Wastes	250-mL amber wide-mouth glass jar or wide mouth plastic bottle	None required	Fill per sampling plan	Analyze as soon as possible. 24 hours maximum	SW-846 Method 9040B, rev 2, 1/95 and Chapter 2, Table 2-33, rev 2, 9/94
Corrosivity to Steel	1-liter plastic jar	Cool to 4°C	Fill per sampling plan	None listed	SW-846 1110, 9/86
Ignitability	250-mL amber wide-mouth glass jar or wide mouth plastic bottle	Cool to 4°C; Non-aqueous wastes should review sampling plan	Fill with no headspace when volatiles are suspected. Check sampling plan.	None listed	SW-846 Method 1010, 9/86; SW-846 Method 1020, Rev 1, 7/92 ASTM D-93-79; D-93-80 ASTM D-3278-78
Reactivity (releasable sulfide and releasable cyanide). *(Note 5)*	Two 250-mL amber wide-mouth glass jars or wide mouth plastic bottle	Cool to 4°C and keep away from light. Review sampling plan.	Fill with no headspace and per sampling plan	Analyze as soon as possible	SW-846 §7.3 Regulatory definition Released Sulfide method 7.3.4.2 Released Cyanide method 7.3.4.1, rev 2, 9/94

SOILS, SLUDGES, AND SOLID WASTE (CERCLA High Hazard Protocol) *(Note 1)*

PARAMETER	SAMPLE CONTAINERS	PRESERVATION	FILLING INSTRUCTIONS	HOLDING TIME	REFERENCE
Refer to Navy IR Program					

PCB WIPE SAMPLES (TSCA) *(Note 1)*

PCBs	4-oz wide-mouth glass jar	None	One wipe pad per jar	Not established	40CFR 761.123 June 19, 1987

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 SDWA Fluoride: Distillation step need only be performed once for each sample location to demonstrate that it is not necessary for that sample matrix and location.

Note 3 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 4°C, reduce chlorine residual with 0.008% sodium thiosulfate, store in dark, adjust the pH to 6-9, and hold for seven 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, and extracts may be stored for 7 days if stored under inert atmosphere. The pH adjustment may be performed upon receipt at the lab or may be omitted if the samples are extracted within 72 hours of collection. For analysis of aldrin, add 0.008% sodium thiosulfate ($Na_2S_2O_3$).

Note 4 Collect sample in 125 mL container which has been preserved with 4 drops of 10% sodium thiosulfate ($Na_2S_2O_3$) solution. Gently swirl to mix and transfer to vial. Acid addition to pH < 2 may be with HCl, H_2SO_4, or solid $NaHSO_4$. Cool.

Note 5 Reactivity: Total analysis for cyanide and sulfide may be substituted if allowed in sampling plan. Preservation for total cyanide and sulfide is per Groundwater: RCRA; Other Waters and Non-aqueous Liquids: RCRA; and Soils, Sludges and Solid Waste: RCRA; tables found above.

Table H-2 Requirements for Biological Sample Preservation

ORGANISMS	PRESERVATION
Fish *(Notes 1,2)*	Refer to sampling plan for project specific requirements. Preservation is dependent upon purpose and species being sampled.
Bottom Associated Organisms *(Note 2)*	Refer to sampling plan for project specific requirements. Preservation is dependent upon purpose and species being sampled.
Algae *(Note 2)*	Refer to sampling plan for project specific requirements. Preservation is dependent upon purpose of the measurement.

Notes:

Note 1 Jenkins, R.E. and N.M. Burkhead. 1993. Freshwater Fishes of Virginia. American Fisheries Society, Bethesda, Maryland.
Note 2 Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992. Other preservation solutions are provided in this reference.

APPENDIX I

REQUIREMENTS FOR COLLECTION OF QUALITY CONTROL SAMPLES

Table I-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 Matrix Spike/Matrix Spike Duplicate (MS/MSD) lab studies, the laboratory shall 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 H**.

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.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE 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 FSP and requested to be the specific matrix sampled. The laboratory may run one MS/MSD of each group of water samples which may not include the exact matrix being 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.

DRINKING WATER (SDWA)

PARAMETERS	**FIELD DUPLICATES**	**TRIP BLANKS**	**EQUIPMENT DECONTAMINATION BLANKS**	**FIELD BLANKS**	**MATRIX SPIKE/MATRIX SPIKE DUPLICATE 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	None *(Note 4)*
Synthetic Organic Chemicals (Pesticides, Herbicides, PCBs)	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None *(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	None *(Note 5)*
Lead and copper	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None *(Note 5)*
Fluoride	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None *(Note 5)*
Silica	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None *(Note 5)*
Orthophosphate	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None *(Note 5)*

DRINKING WATER (Continued)

PARAMETERS	FIELD DUPLICATES	TRIP BLANKS	EQUIPMENT DECONTAMINATION BLANKS	FIELD BLANKS	MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES
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	None *(Note 5)*
Nitrite	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None *(Note 5)*
Radionuclides	None required *(Note 1)*	None	*(Note 8)*	*Note 9* may be performed based on data needs	None

WASTEWATER NPDES PERMIT SAMPLING (CWA)(Check state specific program for QC criteria)					
PARAMETER	**FIELD DUPLICATES**	**TRIP BLANKS**	**EQUIPMENT DECONTAMINATION BLANKS**	**FIELD BLANKS**	**MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES**
Dissolved oxygen	N.A. - Field Test				
Biological 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
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

WASTEWATER NPDES PERMIT SAMPLING (Continued)

PARAMETER	**FIELD DUPLICATES**	**TRIP BLANKS**	**EQUIPMENT DECONTAMINATION BLANKS**	**FIELD BLANKS**	**MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES**
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

GROUNDWATER (RCRA; SW-846, July, 1992, Rev 1)

PARAMETER	**FIELD DUPLICATES**	**TRIP BLANKS**	**EQUIPMENT DECONTAMINATION BLANKS**	**FIELD BLANKS**	**MATRIX SPIKE/MATRIX SPIKE DUPLICATE 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)*

OTHER WATERS AND NON-AQUEOUS LIQUIDS (RCRA; SW-846, July 1992, Rev 1)

PARAMETER	**FIELD DUPLICATES**	**TRIP BLANKS**	**EQUIPMENT DECONTAMINATION BLANKS**	**FIELD BLANKS**	**MATRIX SPIKE/MATRIX SPIKE DUPLICATE 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)*
OTHER WATERS AND NON-AQUEOUS LIQUIDS - Low and Medium Concentration (CERCLA)					
Refer to Navy IR Program					
OTHER WATERS AND NON-AQUEOUS LIQUIDS - High Hazard Protocol (CERCLA)					
Refer to Navy IR Program					

SOILS, SLUDGES, AND SOLID WASTE (RCRA; SW-846, July 1992, Rev 1)

PARAMETER	FIELD DUPLICATES	TRIP BLANKS	EQUIPMENT DECONTAMINATION BLANKS	FIELD BLANKS	MATRIX SPIKE/MATRIX SPIKE DUPLICATE 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)*

SOILS, SLUDGES, AND SOLID WASTE - High Hazard Protocol (CERCLA)

PARAMETER	FIELD DUPLICATES	TRIP BLANKS	EQUIPMENT DECONTAMINATION BLANKS	FIELD BLANKS	MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES
Refer to Navy IR Program					

PCB WIPE SAMPLES (TSCA)

PARAMETER	FIELD DUPLICATES	TRIP BLANKS	EQUIPMENT DECONTAMINATION BLANKS	FIELD BLANKS	MATRIX SPIKE/MATRIX SPIKE DUPLICATE 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 4 sample containers for one sample in 20 for each matrix type per method.
Note 5 Water samples for inorganic analysis:
One additional sample containers 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 1 MS/MSD per matrix per method or 1 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 1 MS/MSD per matrix per method or 1 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 precleaned 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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APPENDIX J

BRIEF DESCRIPTION OF EPA AND OTHER AIR POLLUTION MODELS

APRAC-3	Computes hourly averages of carbon monoxide for urban locations.
BLP	Buoyant Line Plume Model is a Gaussian plume dispersion model for aluminum reduction plants and graphic electrode plants.
CALINE3	Calculates carbon monoxide concentrations near highways and arterial streets.
CALMPROA	Processor for output of short-term models that calculates concentrations for non-calm hours based on recommendations in EPA's *Modeling Guideline*.
CDM-2.0	This climatological dispersion model determines long-term quasi-stable pollutant concentrations.
CHAVGA	Somewhat slower post-processor than RUNAVG that determines the second highest non-overlapping running average.
COMPLEX-1	A multiple point source code with terrain adjustment using sequential meteorological data to calculate concentrations using the VALLEY algorithm.
CRSTER	Estimates ground-level concentrations resulting from up to 19 collated elevated stack emissions. HIWAY2 computes the hourly concentrations of non-reactive pollutants downwind of roadways.
INPUFF	An integrated puff model for analyzing non-continuous accidental releases of substances over a period of minutes to several hours.
ISCLT	Industrial Source Complex Long-Term is a steady-state Gaussian plume model which can be used to calculate long-term pollutant concentrations from an industrial source complex.
ISCST	Industrial Source Complex Short-Term is a steady-state Gaussian plume model which can be used to calculate short-term pollutant concentrations from an industrial source complex.
LONGZ	Designed to calculate the long-term pollutant concentration produced at a large number of receptors by emission from multiple stack, building, and area sources in urban areas.
MESOPUFF-2.0	A variable-trajectory regional-scale Gaussian puff model especially designed to simulate the air quality of multiple point sources at long distances.
MPDA-1.1	A processor of meteorological data for use with TUPOS-2.0.
MPTDS	A modification of MPTER to explicitly account for gravitational settling and/or deposition loss of a pollutant.
MPTER	A multiple point-source Gaussian model with optional terrain adjustments.
PAL-2.0	Point, Area, Line Source Algorithm is a short-term Gaussian steady-state algorithm which estimates concentrations of stable pollutants from point, area, and line sources.

PBM	A simple stationary single-cell numerical model that calculates hourly averages of ozone and other photochemical pollutants.
PEM-2	An EPA-developed modification which includes deposition of the TEM8B model for use in urban areas.
PLUVUE-2	A visibility model designed to predict the transport, atmospheric diffusion, chemical conversion, optical effects and surface deposition of point-source emissions.
PTPLU-2	A point source dispersion Gaussian screening model for estimating maximum surface concentrations for one-hour concentrations.
RAM	Short-term Gaussian steady-state algorithm estimates concentrations of stable pollutants that has been historically applied in urban areas.
RAMMET	Processes hourly meteorological data for use with models such as CRSTER, RAM, ISCST, MPTER, TEM8AB, COMPLEX1, and BLP.
RTDM 3.2	A model offering improvements on COMPLEX1 for calculating concentrations on elevated terrain.
ROADWAY-2.0	A finite difference model which predicts pollutant concentrations near a roadway.
RUNAVG	A post-processor for determining the second-highest non-overlapping running average.
SCREEN	A PC screening model developed by the EPA to analyze stacks, flares and area sources in simple and complex terrain.
SHORTZ	Designed to calculate the short-term pollutant concentration produced at a large number of receptors by emission from multiple stack, building, and area sources in urban areas.
TUPOS-P	The post-processor for TUPOS-2.0
TUPOS-2.0	An improved version of MPTER that estimates dispersion directly from fluctuation statistics at plume level.
UTMCON	A processor that converts UTM coordinates to latitude and longitude and vice-versa.
VALLEY	A steady-state, univariant Gaussian plume dispersion model useful for calculating concentrations on any type of terrain.

The following models are used for air pollution analysis in certain regions of the United States:

TCM2B	Texas Climatological Model is a climatological, Gaussian plume model for determining long-term average pollutant concentrations of non-reactive pollutants.
TEM8AB	Texas Episodic Model is a sequential, steady-stat Gaussian plume model for determining short-term concentrations of non-reactive pollutants.
MPTER	A version of MPTER modified by the New York City Department of Environmental Protection.

Important models available from other sources:

CALINE4	An improved version of CALINE3 that includes emissions from highways, intersections and parking lots.

DEGADIS	A U.S. Coast Guard model that analyzes the effects of dense gas releases and calculates the shape of the area in which concentrations will exceed a user-selected value.
OCD	Offshore and Coastal Dispersion Model employs direct measurement of atmospheric turbulence to calculate on-shore concentrations from off-shore emissions.
MS-PUFF	A modification to MESOPUFF made for North Dakota for use in estimating PSD increments for Class I areas.
SPILLS	A model available from NTIS especially useful for calculating the evaporation rate of substances from a liquid pool and their dispersion in the atmosphere.
TRPUF	A Trinity Consultants, Inc. model employed to study to dispersion of individual puff releases.

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APPENDIX K

EPA EMISSION TEST METHODS UNDER TITLE 40

METHOD	STATUS[1]	PARAMETER
Part 60 Appendix A		
1	F	Sample and velocity traverses for stationary sources
1A	F	Sample and velocity traverses for stationary sources with small stacks or ducts
2	F	Determination of stack gas velocity and volumetric flow rate (Type S pitot tube)
2A	F	irect measurement of gas volume through pipes and small ducts
2B	F	Determination of exhaust gas volume flow rate from gasoline vapor incinerators
2C	F	Determination of stack gas velocity and volumetric flow rate in small stacks or ducts (standard pitot tube)
2D	F	Measurement of gas volumetric flow rates in small pipes and ducts
2E	P	Determination of gas flow rate from landfill wells
2F	T	Use of 3-D pitot tube for gas velocity measurements
3	F	Gas analysis for carbon dioxide, oxygen, excess air, and dry molecular weight
3A	F	Determination of oxygen and carbon dioxide concentrations in emissions from stationary sources (Instrumental Analyzer Procedure)
3B	F	Orsat for correction factors and excess air
3C	P	Determination of gas composition of landfill gases
4	F	Determination of moisture content in stack gases
5	F	Determination of particulate emissions from stationary sources
5A	F	Determination of particulate emissions from the asphalt processing and asphalt roofing industry
5B	F	Determination of non-sulfuric acid particulate matter from stationary sources
5C	T	Determination of particulate matter from small ducts
5D	F	Determination of particulate emissions from positive pressure fabric filters

METHOD	STATUS[1]	PARAMETER
5E	F	Determination of particulate emissions from the wool fiberglass insulation manufacturing industry
5F	F	Determination of nonsulfate particulate matter from stationary sources
5G	F	Determination of particulate emissions from wood heaters from a dilution tunnel sampling location
5H	F	Determination of particulate emissions from wood heaters from a stack location
6	F	Determination of sulfur dioxide emissions from stationary sources
6A	F	Determination of sulfur dioxide, moisture, and carbon dioxide emissions from fossil fuel combustion sources
6B	F	Determination of sulfur dioxide and carbon dioxide daily average emissions from fossil fuel combustion sources
6C	F	Determination of sulfur dioxide emissions from stationary sources (Instrumental Analyzer Procedure)
7	F	Determination of nitrogen oxide emissions from stationary sources
7A	F	Determination of nitrogen oxide emissions from stationary sources - Ion Chromatographic Method
7B	F	Determination of nitrogen oxide emissions from stationary sources (Ultraviolet Spectrophotometry)
7C	F	Determination of nitrogen oxide emissions from stationary sources - (Alkaline-permanganate/colorimetric method)
7D	F	Determination of nitrogen oxide emissions from stationary sources - (Alkaline-permanganate/ion chromatographic method)
7E	F	Determination of nitrogen oxide emissions from stationary sources - (Instrumental Analyzer Procedure)
8	F	Determination of sulfuric acid mist and sulfur dioxide emissions from stationary sources
9	F	Visual determination of the opacity of emissions from stationary sources
9A	F	Visual determination of the opacity of emissions from stationary sources remotely by lidar
10	F	Determination of carbon monoxide emissions from stationary sources
10A	F	Determination of carbon monoxide emissions in certifying continuous emission monitoring systems at petroleum refineries
10B	F	Determination of carbon monoxide emissions from stationary sources
11	F	Determination of hydrogen sulfide content of fuel gas streams in petroleum refineries

METHOD	STATUS[1]	PARAMETER
12	F	Determination of inorganic lead emissions from stationary sources
13A	F	Determination of total fluoride emissions from stationary sources - SPADNS zirconium lake method

13B	F	Determination of total fluoride emissions from stationary sources - Specific ion electrode method
14	F	Determination of fluoride emissions from potroom roof monitors for primary aluminum plants
15	F	Determination of hydrogen sulfide, carbonyl sulfide, and carbon disulfide emissions from stationary sources
15A	F	Determination of total reduced sulfur emissions from sulfur recovery plants in petroleum refineries
16	F	Semicontinuous determination of sulfur emissions from stationary sources
16A	F	Determination of total reduced sulfur emissions from stationary sources (impinger technique)
16B	F	Determination of total reduced sulfur emissions from stationary sources
17	F	Determination of particulate emissions from stationary sources (in-stack filtration method)
18	F	Measurement of gaseous organic compound emissions by gas chromatography
19	F	Determination of sulfur dioxide removal efficiency and particulate, sulfur dioxide and nitrogen oxides emission rates
20	F	Determination of nitrogen oxides, sulfur dioxide, and diluent emissions from stationary gas turbines
21	F	Determination of volatile organic compound leaks
22	F	Visual determination of fugitive emissions from material sources and smoke emissions from flares
23	F	Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans From Stationary Sources
24	F	Determination of volatile matter content, water content, density, volume solids, and weight solids of surface coatings
24A	F	Determination of volatile matter content and density of printing inks and related coatings
25	F	Determination of total gaseous non-methane organic emissions as carbon
25A	F	Determination of total gaseous organic concentration using a flame ionization analyzer
25B	F	Determination of total gaseous organic concentration using a non-dispersive infrared analyzer

METHOD	**STATUS[1]**	**PARAMETER**
25C	P	Determination of volatile organic compounds from landfills
25D	F	Determination of volatile organics from TSDF - purge procedure
25E	F	Determination of volatile organics from TSDF - vapor pressure procedure
26	F	Determination of Hydrogen Chloride Emissions from Stationary Sources
26A	F	Isokinetic determination of hydrogen chlodie, halogens, and other hydrogen halides from stationary sources

27	F	Determination of vapor tightness of gasoline delivery tank using pressure vacuum test
28	F	Certification and auditing of wood heaters
28A	F	Measurement of air to fuel ratio and minimum achievable burn rates for wood-fired appliances
29	P	Determination of multiple metals

Part 60 Appendix B

PS-1	F	Specifications and test procedures for opacity continuous emission monitoring systems in stationary sources
PS-2	F	Specifications and test procedures for SO_2 and NO_x continuous emission monitoring systems in stationary sources
PS-3	F	Specifications and test procedures for O_2 and CO_2 continuous emission monitoring systems in stationary sources
PS-4	F	Specifications and test procedures for carbon monoxide continuous emission monitoring systems in stationary sources
PS-4A	F	Specifications and test procedures for carbon monoxide continuous emission monitoring systems in stationary sources
PS-5	F	Specifications and test procedures for TRS continuous emission monitoring systems in stationary sources
PS-6	F	Specifications and test procedures for continuous emission rate monitoring systems in stationary sources
PS-7	F	Specifications and test procedures for hydrogen sulfide continuous emission monitoring systems in stationary sources
PS-8	F	Specifications and test procedures for volatile organic compounds continuous emission monitoring systems
PS-9	F	Specifications and test procedures for gas chromatographic continuous emission monitoring systems
PS-10	T	Specifications and test procedures for ammonia continuous emission monitoring systems

METHOD	**STATUS[1]**	**PARAMETER**
PS-11	T	Specifications and test procedures for particulate material continuous emission monitoring systems
PS-12	T	Specifications and test procedures for mercury continuous emission monitoring systems

Part 60 Appendix F

PRC 1	F	Quality Assurance Requirements for Gas Continuous Emission Monitoring Systems Used for Compliance Determination

Part 60 Appendix J

APP-J	F	Determination of wood stove thermal efficiencies

Part 61 Appendix B

101	F	Determination of particulate and gaseous mercury emissions from chlor-alkali plants - air streams
101A	F	Determination of particulate and gaseous mercury emissions from sewage sludge incinerators
102	F	Determination of particulate and gaseous mercury emissions from chlor-alkali plants - hydrogen streams
103	F	Beryllium screening method
104	F	Determination of beryllium emissions from stationary sources
105	F	Determination of mercury in wastewater treatment plant sewage sludge
106	F	Determination of vinyl chloride from stationary sources
107	F	Determination of vinyl chloride content of in process wastewater samples, and vinyl chloride content of polyvinyl chloride resin, slurry, wet cake, and latex samples
107A	F	Determination of vinyl chloride content of solvents, resin-solvent solution, polyvinyl chloride resin, resin slurry, wet resin, and latex samples
108	F	Determination of particulate and gaseous arsenic emissions
108A	F	Determination of arsenic content in ore samples from non-ferrous smelters
108B	F	Determination of arsenic content in ore samples from non-ferrous smelters
108C	F	Determination of arsenic content in ore samples from non-ferrous smelters
111	F	Determination of Polonium-210 emissions from stationary sources
114	P	Determination of radionuclides

METHOD	STATUS[1]	PARAMETER
115	P	Determination of Radon-222

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METHOD	STATUS[1]	PARAMETER
201	F	Determination of PM_{10} Emissions (Exhaust Gas Recycle Procedure)
201A	F	Determination of PM_{10} Emissions (CSR Procedure)
202	F	Determination of Condensible Particulate Emissions from Stationary Sources
203	P	Use of Transmissometer for Visible Emissions Measurements for Compliance
203A	P	Visible Emissions Measurements (2 - 6 Minute Averages)
203B	P	Visible Emissions Measurements (Time Exception)
203C	P	Visible Emissions Measurements (Instantaneous)
204	T	Measurement of Volatile Organics Capture Efficiency
204A	T	Measurement of Volatile Organics Capture Efficiency
204B	T	Measurement of Volatile Organics Capture Efficiency
204C	T	Measurement of Volatile Organics Capture Efficiency
204D	T	Measurement of Volatile Organics Capture Efficiency
204E	T	Measurement of Volatile Organics Capture Efficiency
204F	T	Measurement of Volatile Organics Capture Efficiency
205	F	Verification of Gas Dilution Systems for Field Instrument Calibrations
206	T	Determination of Ammonia
207	T	Determination of Isocyanates

Part 63 Appendix A

METHOD	STATUS[1]	PARAMETER
301	F	Field Validation of Pollutant Measurement Methods from Various Waste Media
303	F	Determination of Coke Oven Door Emissions
304A	F	Determination of Biodegradation Rate (Vented)
304B	F	Determination of Biodegradation Rate (Enclosed)
305	F	Compound Specific Liquid Waste

METHOD	STATUS[1]	PARAMETER
306	F	Determination of Chromium Emission from Decorative and Hard Chromium Electroplating and Anodizing Operations

306A	F	Simplified Chromium Emission Sampling
306B	F	Surface Tension Measurement and Recordkeeping for Chromium Plating Tanks
307	F	Determination of Solvent Degreaser Volatile Organic Compounds
308	P	Determination of Methanol Emissions
309	P	Aerospace Solvent Recovery Material Balance
310	T	Determination of Residual Hexane Emissions in EPDM Rubber
311	P	Determination of Emissions of Volatile Organic Compound Hazardous Air Pollutants in Furniture Coatings
312	T	Determination of Residual Styrene Emissions in SBR Rubber
313	T	Determination of Residual Styrene Emissions in PBR Rubber
314	T	Determination of Halogenated Compound Emissions in Solvents
315	T	Determination of Methylene Chloride Extractable Matter
316	T	Determination of Formaldehyde Emissions (Manual Method)
317	T	Determination of Phenol Emissions (Manual Method)
318	T	Determination of Formaldehyde, Phenol, and Methanol by Fourier Transform Infrared Spectroscopy (FTIR)
319	T	Determination of Filter Efficiency for Paint Overspray

Note:

1 Method Status Code:
F = Finalized Methods (Codified)
P = Proposed Method
T = Tentative Method, Under Evaluation

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

APPENDIX L

SUGGESTED TEXT FOR LABORATORY CONTRACTING DOCUMENTATION

NOTE:

The following sample text was taken from a water analysis contract. It is provided as an aid to the reader and offers some things to consider when drafting contractual documentation for laboratory services. Accordingly, it will not be applicable nor complete in all circumstances.

L.1 Scope of Work. The contractor shall provide laboratory services including the furnishing of all labor, transportation, materials, equipment, supplies, facilities, utilities, supervision, quality assurance and quality control required for the pickup, laboratory analysis and reporting of test data regarding water and wastewater samples submitted by __(insert customer name)__.

NOTE:

Other samples such as non-aqueous liquids, solids, sludges and sediments, must incorporate the program and matrix specific regulations for those samples in place of the water and wastewater references cited in this contract.

The unit price for each test method shall include the cost of providing sample containers, performing laboratory analysis per the specified method and reporting of the analysis data for each sample in a report. The unit price will include the furnishing of all labor, materials, equipment, supplies, facilities, utilities and supervision, and all costs associated with the performance of the work specified, including disposal of any unused sample and sample bottles and containers. Unused sample volumes will not be disposed of until thirty (30) days following submission of analysis data in a written report.

Support personnel, test equipment, consumable chemicals and materials, etc., for the performance of the specified tests are the responsibility of the contractor and shall be suitable to satisfy the quality of data required and measure the parameter cited. All equipment and procedures used for the performance of the laboratory analyses shall be in conformance with the regulatory program guidelines cited by the EPA and state program agency. The contractor shall follow and meet the quality control and assurance requirements for the performance of the tests (where available) as stated in the referenced test method and regulatory program documents.

L.2 Specifications and Standards. All laboratory analyses of wastewater and drinking water and other aqueous (water based liquid) samples shall be performed in accordance with the most recent applicable regulations found in the Code of Federal Regulations (CFR), state regulations and guidelines.

NOTE:

Explicitly list the applicable standards and the test parameters. For example, any of the following could be used to define the specifications and standards for this contract:

1) Wastewater testing to comply with 40 CFR Part 136 for the following tests:

Fecal Coliform
Biochemical Oxygen Demand
Total Suspended Solids
Hexavalent Chromium

2) Drinking water testing to comply with 40 CFR Part 141 for the following tests:

Distribution Water
Total Coliform, presence/absence
Total Trihalomethanes

3) Methods contained in Standard Methods for the Examination of Water and Wastewater, 18th edition, to include the quality control cited in Part 1020B, for the following tests:

Total Organic Carbon
Total Kjeldahl Nitrogen
Volatile Suspended Solids
Percent Solids
Ammonia Nitrogen
Nitrate/nitrite
Total Phosphorus

NOTE:

Any data quality objectives (DQO) that are part of the project should also be included as part of the contract to ensure the laboratory performs per the project required DQO.

L.3 Permits and Certifications. The contractor and any/all proposed subcontractors shall have all appointments, licenses and permits required for the completion of the work and for complying with all applicable Federal, state and local laws. All work and services shall be performed by a laboratory which is certified to perform the specified analyses in the state of _(insert State)_ where such certification exists.

NOTE:

> The last sentence "..performed by a laboratory which is certified to perform the specified analysis in the state of _(insert State)_ where such certification exists." usually applies only to drinking water and occasionally wastewater or waste. The following paragraph would supersede this sentence in those states that do not have an appropriate lab certification program or if this contract is used for other types of samples.

If no EPA, state or local certification exists for the program or matrix, a recognized third party accreditation for the test method required will be accepted as demonstration of performance for the test method and matrix. In lieu of certification or accreditation, the Contracting Officer may require an audit of the facility, submission of proficiency testing data and other proofs of compliance with the test methods and program requirements needed for performance of the scope of work to the specification and standards cited.

Throughout the performance of the contract, the customer shall be notified by the prime contractor of any intentions to either add new subcontractors or change any subcontractors previously identified within ten (10) days prior to the anticipated addition or change. The Contracting Officer must approve any substitutions. The customer shall be provided with all proofs of compliance as described in this section for the prime contractor and subcontractors prior to award of the contract and proposed new subcontractors prior to their approval.

L.4 Chain of Custody. A signature and time audit trail (chain of custody) for each sample from the time of collection, to the completion of the analysis, and ultimately, to sample disposal, is required. The chain of custody form shall be developed by the contractor and shall include provisions to ensure that the parties involved sign the form and indicate the time and date the transfer of the sample(s) occurred (each time possession of an individual sample or group is transferred). The chain of custody shall also include a space to indicate if the samples were iced or refrigerated when they were received by the laboratory (for samples that require it). The chain of custody shall include a space for indicating the pH after preservation in the field and indicating the pH when received by the laboratory for samples requiring acid or base preservation. Any discrepancies in cooling and preservation must be indicated and duly reported.

A copy of the chain of custody shall be made after laboratory personnel have signed off on the form. This copy shall remain at the sample pickup site after the samples are picked up, the original form is to accompany the samples.

The anticipated disposal date and method of sample disposal shall be indicated on the chain of custody form. The sample disposal date for unused samples will be thirty (30) days after final report submission unless directed in writing to retain for an extended period of time. The method of disposal must be in accordance with the hazards identified by the customer and determined by the laboratory. The customer will notify the laboratory prior to sample submittal and on the chain of custody any suspected hazardous substances or radioactive material.

The original completed form shall accompany the test results. Chain of custody records shall be maintained by the laboratory for three (3) years for all samples for which test data is required.

L.5 Quality Control Documentation. The contractor is required to have a written description of the Quality Assurance/Quality Control (QA/QC) program that is to be followed by the laboratory throughout the contract period. The QA/QC program should cover laboratory equipment, facilities and personnel, training of personnel, calibration plans, reagent quality, preparation and control of standards, test methods, sample handling, method verification, the use of control and reference samples, evaluation and documentation of data quality and any other policies and plans that will be used by the contractor to ensure the reliability of the laboratory test results. The laboratory chain of custody procedures and record form shall be included as part of the QA/QC program to be submitted by the contractor with the proposal package.

Quality assurance reference samples from the EPA or a qualified third party vendor will be provided through the customer to the laboratory for analysis of the parameters included as part of this contract. At least one (1) set per year will be provided. These samples must be analyzed using the same personnel, equipment, and test methods used for each of the parameters reported during the year. Analysis of these samples and any others required by the EPA or other regulatory agency will be considered part of the contractor's quality control program for this contract. Laboratory analyses for these samples shall be the responsibility of the contractor, and not a cost associated with this contract.

The laboratory shall furnish all available information on the results of EPA performance evaluation samples, intra-laboratory samples, or other analysis of unknown samples to demonstrate proficiency for the parameters listed in the contract. Accuracy of analysis of unknown QA/QC reference samples shall be considered prior to award of this contract. Results of these analyses from previous years, their actual values, and the range of values considered acceptable by the EPA, state agency or third party providing the unknowns, shall be included as part of the QA/QC program to be submitted by the contractor with the proposal package.

L.6 Personnel Qualifications. The laboratory shall be under the supervision of a qualified chemist or scientist, capable of interpreting and directing the analyses to be performed under this contract, and who shall review and certify, by a signed statement on the report form, the adequacy and accuracy of procedures, test results and reports. Personnel performing testing duties shall have demonstrated current experience and proficiency in the methods and procedures required by this contract and this shall be documented in accordance with the laboratory's quality assurance procedures. The chemists, microbiologists and analysts shall have training and experience as described under COMAR 26.08.05 which will qualify them to perform services under this contract. The chemists and microbiologists personnel are considered KEY and are subject to the terms of the key personnel provisions.

L.6.1 Chemical Analyst.

- Supervisory. The water quality laboratory supervisor shall have at least a bachelor's degree in chemistry or in a closely related field with at least two (2) years experience in the performance of chemical tests to be performed under this contract.
- Non-Supervisory. The analysts shall have at least a high school diploma or its equivalent, and at least three (3) months of on-the-job training in the measurement of inorganic or organic parameters for analyses to be performed under this contract.

L.6.2 Microbiological Analyst.

- Supervisory. The water quality laboratory supervisor shall have at least a bachelor's degree in microbiology or in a closely related field with at least two (2) years experience in the performance of microbiological tests to be performed under this contract.
- Non-Supervisory. The analysts shall have at least a high school diploma or its equivalent, and at least three (3) months of on-the-job training in the performance of microbiological tests to be performed under this contract.

L.7 Test Methods. Laboratory analyses shall be performed following the criteria, methods and procedures approved by the U.S. EPA for each type of sample, as stated in 40 CFR part 136, 40 CFR part 141, 40 CFR part 143, 40 CFR part 261 and any subsequent amendments or regulations. Analysis of any parameter not included under the above referenced documents or other EPA approved documents shall be performed in accordance with the approved documents listed under Specifications and Standards.

L.8 Holding Times. The elapsed time from sample collection to the time the analysis is performed is the holding time. **By law, these holding times must not be exceeded when performing testing per the CFR requirements.** The contractor shall be responsible for meeting the holding time required by the type of analysis to be performed. The required holding time shall be in accordance with applicable regulations or guidelines. For example, the maximum holding time for a fecal coliform sample is only six (6) hours when complying with NPDES 40 CFR part 136 test requirements. This should be considered by laboratories several hours away. Traffic or weather conditions are not allowable reasons for exceeding the maximum holding time on a sample.

L.9 Recordkeeping. The contractor will record data required in accordance with the reference methods and regulatory program documents. Records will be maintained showing equipment calibration, standard preparation, traceability of samples, measurements and all information required by the contractor quality assurance plan and regulatory requirements. The contractor will maintain records for at least three (3) years or as otherwise required by the appropriate permits. These records and contractor facilities are subject to inspection at any reasonable time by the EPA, state or Navy personnel.

L.10 Sample Transportation. Samples that require refrigeration shall be transported by the contractor in an iced cooler provided by the contractor. Samples shall be transported to the laboratory for analysis by the contractor.

Samples will be collected by Government personnel on a routine and non-routine basis as described below. Charges for routine and non-routine pickups shall be included in the cost estimate.

NOTE:

> Include any specific information on sample transportation and handling. Specific information on preservation, holding time, containers and test methods may be found in **Appendix H**. The sample handling process must thoroughly describe the proper handling

and adequate transportation to ensure that the sample integrity is not compromised.

- Routine Pickup. The contractor shall pick up samples twice a week from a central location as specified by the customer. Samples shall be picked up on Wednesdays and Fridays between 12 noon and 1:00 p.m.

- Non-routine Pickup. Some samples shall be picked up on non-pickup days due to irregular processing schedules and short (less than forty eight [48] hour) holding times. The average is about three (3) non-routine pickups per month. The laboratory will only be given 24 hour notice for these pickups. The pickups will be at 4:00 pm the day after the contractor is notified of the need for a pickup.

The contractor shall provide a plan for routine and non-routine pickups that shows how the contractor plans to meet short holding times based on travel time to the laboratory, laboratory hours, etc. The plan should be based on the following worst case (shortest holding time) examples:

- Example 1. All wastewater fecal coliform samples will be routine pickups. Since these samples have a six (6) hour holding time, every effort is made to take the sample close to the pickup time so the laboratory has the maximum time to analyze the sample. The earliest the samples are taken is 10:00 am on pickup days, giving the laboratory until 4:00 pm on that day to begin analysis (only three (3) to four (4) hours from the scheduled routine pickup time).

- Example 2. Non-routine pickups may be for hexavalent chromium samples, they have a twenty four (24) hour holding time. These samples will be collected between 8:00 am and 3:00 pm and will be ready for pickup at 4:00 pm the day they are collected. The worst case would be that a contractor would have to pickup a sample at 4:00 pm and start the analysis before 8:00 am the next morning for samples that were collected at 8:00 am on pickup day. (On days when an early sample is collected and no samples are to be collected that afternoon, the laboratory may pick the samples up earlier.)

L.11 Telephone Reports. Records of telephone reports shall be maintained as part of the record keeping process of the laboratory. Records shall include date, time, Navy person contacted, contractor personnel, and the information transmitted.

The contractor shall report to the customer by telephone in the following cases:

- Fast Response Requested. Telephone reports shall be made as soon as analysis is completed if a fast analysis response is requested. The telephone report shall include the test result, units, other data required for reporting per the test method and any abnormal or deviations noted.

- Abnormal Sample Condition. The contractor shall immediately report by telephone when the following conditions are observed:
  - The sample shows a lack of homogeneity (i.e. sample has excessive sediment, excessive amount of floating material, or physical appearance is markedly different from that observed for any four (4) previous samples collected from that sampling point.)
  - The sample has an abnormal odor.
  - The sample has an abnormal color.
  - The sample shows other abnormal conditions.

- Abnormal Results Revealed During Analysis. The contractor shall make same day telephone reports whenever any analysis result is:
  - An exception to NPDES permit conditions
  - A positive result for coliform bacteria in drinking water samples
  - A violation of the total trihalomethanes maximum contaminant levels for drinking water samples
  - Matrix quality control is outside expected limits

L.12 Written Reports. The test results submittal shall be in typed form and shall include the following information:

- Sample identification (sample numbers and descriptions provided by the customer with the sample shall be used as identification)
- Dates and times analyses performed

- Name of analyst(s) who performed each analysis (initials acceptable)
- Parameters analyzed
- Analytical and microbiological techniques or methods used
- Results of all required analyses reported with units of measure and other reporting data required by the test method
- Report on deviations or abnormalities, if applicable
- Complete chain of custody form
- Laboratory supervisor signature certifying the adequacy and accuracy of procedures and test results

The contractor shall complete and deliver written reports for analyses of sanitary wastewater samples within ten (10) calendar days of the date samples are picked up by the contractor. The parameters will include fecal coliform, total suspended solids, volatile suspended solids, percent solids, total Kjeldahl nitrogen, ammonia nitrogen, nitrate/nitrite, total phosphorous and biochemical oxygen demand samples.

The contractor shall complete and deliver written reports for all other samples by the tenth (10) of the month following the month in which the samples were collected.

The contractor shall submit a corrected written report, at no additional charge to the Government, if the original report contains any errors or omits any of the required information. The corrected report shall be delivered within five (5) days of notification or discovery of the error or omission. The corrected report will be so identified and include the reason for the correction.

When the contractor must repeat any analysis due to problems of contractor origin the analysis shall be completed and submitted to the government within seventy two (72) hours of having to repeat the analysis. (i.e., casting doubt on analysis due to quality control (QC) problems or incorrect test methods.)

L.13 Payment. The laboratory will be paid for results received in the proper report format for samples with a valid chain of custody that have not exceeded their holding times (as stated in CFR) and were tested with the proper test method showing valid quality control data. Results on an incomplete report, not meeting holding time requirements or for a sample that does not have a chain of custody will be declared invalid. Results reported showing the test method meets all method specified control criteria for blanks, laboratory control standards and other quality control samples, except for control criteria for the specific customer matrix will be paid. The government will not pay for any repeated analyses resulting from laboratory error.

L.14 Laboratory information to be collected prior to contract award.

- Results of EPA performance evaluation samples, intralaboratory samples, or other analyses that demonstrate the laboratory's proficiency for analyzing unknown performance evaluation samples in the matrix and for the parameters included in the scope of work (i.e. EPA DMR-QA Study results or similar studies)
- Resumes of key personnel
- A copy of the laboratory's chain of custody form
- A copy of the laboratory's plan for routine and non-routine pickups that shows how the contractor plans to meet holding times based on travel time, laboratory hours, and sample handling procedures to ensure holding times will be met
- A copy of the report format that will be used
- A brief discussion of the procedure established to handle telephone reports
- A notarized copy of the laboratory's certification specifying the categories, parameters and test methods within each category for which the laboratory is certified or other accreditation certificate with parameters and categories or signed audit and proficiency test data from EPA, state or Navy personnel for parameters and categories in the scope of work
- A copy of the laboratory's current quality assurance documentation which describes the use of quality control samples and reference samples; preparation and control of standards; calibration of equipment; training of personnel; verification of personnel competency; traceability of samples, measurements and test methods; safety and chemical hygiene plan, waste disposal plan including samples and containers, and reference to in-house test procedures or reference test methods
- Information regarding the use of, and percentage of use of, subcontractor(s) and proof that the contractor and any proposed subcontractor(s) have obtained all required appointments, licenses and permits and complies with all requirements under Quality Assurance and Quality Control
- The plan or procedure for staying abreast of all rules/standards for performance of the parameters in the scope of work (i.e. computer database, library, publications (e.g. Federal Register)

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

APPENDIX M

REQUEST FORM FOR PROPOSED CHANGES TO THE ENVIRONMENTAL SAMPLING PROCEDURES MANUAL

Proposed changes to this manual shall be submitted using the Environmental Sampling Procedures Manual Change Request (MCR) Form, **Figure M-1**. Change requests shall include specific recommended change wording, with detailed rationale or justification. Change requests shall also identify known impacts of the recommended change upon related reference documentation. Completed forms should be submitted directly to NAVSEA 07Q(LABS) at the following address:

NAVSEA 07Q(LABS)
Attn: Sampling Manual
3601 Meeting Street Road
Charleston, SC 29405-5733

Forms (locally generated, with the same information) may be transmitted electronically to:

darley_skip@hq.navsea.navy.mil

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MANUAL CHANGE REQUEST (MCR) FORM

MANUAL REVISION/CHANGE AGAINST WHICH RECOMMENDED CHANGE IS WRITTEN:	ORIGINATING ACTIVITY: (ADDRESS)
PRIMARY MANUAL CHAPTER/SECTION/PARAGRAPH IMPACTED BY RECOMMENDED CHANGE:	
OTHER MANUAL SECTIONS AFFECTED:	POINT OF CONTACT:

RECOMMENDED CHANGE:

RATIONALE/JUSTIFICATION:

RELATED DOCUMENTATION: (This includes any references or documents which may be affected by recommended change).

ORIGINATOR'S SIGNATURE:	DATE:

Figure M-1 Manual Change Request Form
(This Form May Be Reproduced Locally)

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NAVY ENVIRONMENTAL COMPLIANCE SAMPLING & FIELD TESTING PROCEDURES MANUAL

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 in the lead and copper rule.
Active Sampler	In air monitoring context, a sampling device which 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 which results in severe biological harm or death in a short period of time.
Adiabatic	Pertaining to a thermodynamic process executed at constant entropy. No external heating or cooling is artificially introduced to the process under study, the only temperature changes result from environmental temperature and atmospheric pressure.
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 micron particle size range.
Amberlite	XAD-2 resin is 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 which 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 which 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 which 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 which 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 which 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	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 clean up 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. to 200° F.
Community Water System (CWS)	A public water system which 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 which 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 some 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, 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 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.
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 means chart for standards, matrix spikes, calibration check standards, laboratory control standards, means chart for background or blank results and a range chart for replicate analysis.
Coroliolis Force	A fictitious force used mathematically to describe the motion, as of aircraft, cloud formations and air masses, relative to a noninertial, 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, metals or burns, irritates or destructively attacks organic tissue. See 40 CFR Part 264 for definition of Corrosivity under RCRA.

Conventional Pollutants	In the 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.
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 which 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.
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 which 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 (greater than 1) Conductivity (µmhos/cm @ 25° C.) less than 0.1 (equal to 1) Total oxidizable organic carbon (mg/liter) less than 0.05 (less than 0.2) Total solids (mg/liter) less than or equal to 0.1

	(equal to 1) SiO_2 (mg/liter) less than 0.05 (less than 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.
Diethylphenylenediamine (DPD)	The reagent used in a colorimetric test to determine residual free chlorine (disinfectant) concentrations.
Dry Adiabatic Lapse Rate	The rate of temperature change as a dry air mass rises or falls, assuming no addition of heating or cooling. Near the earth's surface, the value is approximately 0.0098^{o} C. per meter.
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 which 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, ground water, 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 which 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 nonoccupational 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 a 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% or less (by volume) with air forms a flammable mixture or the flammable range with air is wider than 12% regardless of the lower limit (at normal temperature and pressure). (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 micron diameter.
Follow-up Monitoring	Testing defined in the drinking water program (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 self contained breathing apparatus (SCBA).
Fume	Fume aerosol particulates from condensation of distillation vapors, calcination, or chemical reactions.
Fungicide	A chemical that prevents the growth of fungus and whose use is approved 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 through a vacuum and 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 separation and then the characteristic ion patters are detected by a mass spectrometer. 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 which is collected at one point in time and place.
Gradient Wind	Free blowing wind above the boundary layer of the atmosphere.
Graphic Furnace (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.
Ground Water	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.
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.
Hazard Class	A category of hazard associated with an 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 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 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.
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 exceed 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 and through final closure.
Health and Safety Plan	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.
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.
In Situ	Within place; commonly used to refer to the location of activities (e.g., in situ soil vapor measurements).
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.

J

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
Lower Flammable Limit (LFL)	The concentration of a chemical in air over a liquid that has been heated to its flash point
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.

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 matrix spike duplicate. The matrix sample, matrix spike and matrix spike duplicate 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 which are very critical to air sampling and monitoring activities.
Method	A sampling or measurement method which has bee 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	The minimum concentration of an analyte in a specific matrix and with a specific method, is measured and reported with a 99% confidence that the concentration of the analyte is greater than zero.
Micro-organism	An organism of microscopic or ultramicroscopic size.
Milligram	A metric unit of weight. There are 1000 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 six 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), that 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 which 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 which 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 eight 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 8 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 a 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.
$PM_{2.5}$	Particles having an aerodynamic diameter less than or equal to 2.5 micrometers.
PM_{10}	Particles having an aerodynamic diameter less than or equal to 10 micrometers.
Point Source	In air sampling, a fixed emission source such as a stack, vent, or discharge pipe.
Point Sources (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 discharged. 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 LD_{50} of up to and including 50 mg/kg, or in inhalation LC_{50} of up to and including 2 mg/liter, or a general LD_{50} 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 fifteen 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 which 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 reconstructions and evaluation of the reports of that study. Raw data may include photographs, 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 which 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 the 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 which 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 Compounds (SOC)	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 which 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 man-made origin (0.01 - 1.0 micron diameter).
Sorbent	A solid material used to extract a chemical contaminant from a gas or a liquid.
Subsurface Soil	The soil more than a defined distance below grade and extending downward to the top of the seasonally high water table.
Split Spoon Sampler	Soil sampling device used during drilling of boreholes to obtain soil samples at specific depths.
Standard Operating Procedure	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 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.
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 man-made 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.
Tenax7	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% of the final response to an incremental change in contaminant concentration.

Trace Analyte	The trace metal(s) or trace organic(s) which are to be determined in the sample of interest. See **Table 13-1** for a listing of typical trace metals of interest.
Traceability	The property of a sample or measurement whereby it can be related to appropriate international or national standards through an unbroken chain.
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 Metals	Naturally occurring chemical elements which 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 (See **Chapter 13, Table 13-1**).
Trace Metal Grade	Reagents which have demonstrated to be free from the metal(s) of interest at the method detection limit (MDL) of the analytical method to be used.
Trace Organics	Man-made (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 man-made compounds such as dioxins and furans.
Transient Non-community Water Systems (TNCWS)	A non-community water system that does not regularly serve at least 25 of the same persons over six 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®	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 Compounds (VOC)	Organic compounds that have a vapor pressure range $>10^{-2}$kPa.
Volatilization	Process whereby solids and liquids pass into the vapor state at a given temperature.

W

Waiver	A document that relinquishes a 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 ground water before a sample is collected.
Wet Collectors	In air sampling context, a collection device which uses a finely dispersed liquid to increase the size of aerosol particles.
Wetland	Those areas that are inundated or saturated by surface or ground water 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.

X

Y

Z

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ACRONYMS

A

ACM	Asbestos Containing Material
ACO	Administrative Consent Order
AL	Action Level
ANSI	American National Standards Institute
AOC	Area of Concern
APA	Air Pathway Analysis
APPS	Act to Prevent Pollution from Ships
APR	Air Purifying Respirator
ARARs	Applicable or Relevant and Appropriate Requirements
AST	Aboveground Storage Tank
ASTM	American Society of Testing and Materials

B

BA	Biological Assessment
BAT	Best Available Technology
BATEA	Best Available Technology Economically Available
BCT	Best Conventional Technology
BDAT	Best Demonstrated Available Technology
BOD	Biochemical (or Biological) Oxygen Demand
BTEX	Benzene, toluene, ethylbenzene, and xylenes

C

CAA	Clean Air Act
CAA90	Clean Air Act Amendments of 1990
cc	Cubic centimeter. A volume measurement in the metric system equal to one milliliter (mL). One quart is about 946 cubic centimeters.
CEMS	Continuous Emission Monitoring System

CERCLA	Comprehensive Environmental Response, Compensation and Liability Act (1980) ("Superfund")
CFC	Chlorofluorocarbon
CFR	Code of Federal Regulations
CGA	Combustible Gas Analyzer
CLP	EPA Contract Laboratory Program
COC	Chain of Custody
COD	Chemical Oxygen Demand
COLIWASA	Composite Liquid Waste Sampler
CPR	Cardiopulmonary resuscitation
CPSC	Consumer Products Safety Commission
CWA	Clean Water Act
CWS	Community Water System

D

DL	Detection Limit
DNAPL	Dense Non-Aqueous Phase Liquid
DNPH	2,4-Dinitrophenyl Hydrazine
DO	Dissolved Oxygen
DOD	Department of Defense
DON	Department of Navy
DOT	Department of Transportation
DOW	Depth of Well
DPD	Diethylphenylenediamine
DQOs	Data Quality Objectives
DRE	Destruction and Removal Efficiency (performance requirement for hazardous waste incinerators
DTW	Depth to Water

E

ECD	Electron capture detector - used in gas chromatography to detect chlorinated chemicals
ECRA	Environmental Cleanup Responsibility Act
EIS	Environmental Impact Statement
EKG	Electrocardiogram
EMMC	Environmental Monitoring Management Council
EOD	Explosive Ordnance Disposal
EP	Extraction Procedure
EPA	United States Environmental Protection Agency
ESLI	End of Service Life
eV	Electron volt - measure of the energy of light having a certain color or frequency

F

FAA	Federal Aviation Administration
FFCA	Federal Facility Compliance Act
FHSLA (CPSC)	Federal Hazardous Substance Labeling Act (See Title 15 USC 1261-1275)
FID	Flame ionization detector. Detector used in real time air monitoring instrument that uses a hydrogen flame to ionize chemicals
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act (See Title 40 CFR)
FM	Factory Mutual
FP or fl. pt	Flashpoint
FPD	Flame Photometric Detector
FS	Feasibility Study
FSP	Field Sampling Plan
FWPCA	Federal Water Pollution Control Act (1972)

G

g	Gram
GC	Gas chromatograph - instrument used to separate mixtures of organic chemicals that can be vaporized without degrading
GIS	Geographic Information System
GPR	Ground Penetrating Radar

H

HAPs	Hazardous Air Pollutants
HASP	Health and Safety Plan
HEPA	High efficiency particulate filter
Hg	Mercury - volatile inert metal
HI	Hazard Index (for noncarcinogens)
HIS	Hazard Information System
HMIS	Hazardous Materials Information System
HMTA	Hazardous Materials Transportation Act (1975)
HNU	Trade name for a portable photoionization air monitoring instrument
HOC	Halogenated Organic Compounds
HPLC	High performance liquid chromatography - instrument used to separate mixtures of organic chemicals that are present in a liquid solution
HRGC/HRMS	High Resolution Gas Chromatography/High Resolution Mass Spectrometry
HSL	Hazardous Substance List
HSO	Health and Safety Officer
HSWA	Hazardous and Solid Waste Amendments of 1984 (RCRA Jr.)
HWS	Hazardous Waste Sites

I

IATA	International Air Transport Association
ICAO	International Civil Aviation Organization
ICP	Inductively Coupled Plasma
ID	Infrared Detector
IDLH	Immediately Dangerous to Life and Health
IR	Installation Restoration

J

K

kg	Kilogram
kPa	Kilopascals

L

LEL	Lower Explosive Limit
LFL	Lower Flammable Limit
LNAPL	Light Non-Aqueous Phase Liquid
LUST	Leaking Underground Storage Tanks

M

MCL	Maximum Contaminant Level
MCLG	Maximum Contaminant Level Goal
MDL	Method Detection Limit
mg	Milligram
ML	Minimum (Reporting) Level
mL	Milliliter
MSDS	Material Safety Data Sheet
MSHA	Mine Safety and Health Administration
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSW	Municipal Solid Waste
MWC	Municipal Waste Combustor

N

NAAQS	National Ambient Air Quality Standards
NAPL	Non-Aqueous Phase Liquid
NAVOSH	Naval Occupational Safety and Health
NBS	National Bureau of Standards
NCDC	National Climatic Data Center
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NEPA	National Environmental Protection Act

NESHAPs	National Emission Standards for Hazardous Air Pollutants
NFPA	National Fire Protection Association
NIOSH	National Institute of Occupational Safety and Health
NIPDWR	National Interim Primary Drinking Water Regulations
NIST	National Institute of Standards
NMOC	Non-Methane Organic Compounds
NOAA	National Oceanic and Atmospheric Administration
n.o.s.	Not Otherwise Specified
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities Site List
NSPS	New Source Performance Standards
NSR	New Source Review
NTIS	National Technical Information Service
NTNCWS	Non-transient Non-community Water Systems
NTU	Nephelometric Turbidity Unit

O

OPNAV	Office of the Chief of Naval Operations
OPNAVINST	Office of the Chief of Naval Operations Instruction
ORME	Other Regulated Materials
OSHA	Occupational Safety and Health Administration or Occupational Safety and Health Act
OSWER	Office of Solid Waste and Emergency Response (EPA)
OVA	Organic Vapor Analyzer, trade name of a portable flame ionization detector (FID) produced by Century, Inc.
OVM	Organic Vapor Meter, trade name of a portable photoionization detector (PID) produced by Termo-Environmental, Inc.

P

PA	Pollution Abatement
P/A	Presence or Absence

PA/SI	Preliminary Assessment/Site Inspection
PAHs	Polynuclear Aromatic Hydrocarbons
PANs	Peroxycarboxyclic Nitric Anhydrides
PCBs	Polychlorinated biphenyls
PCE	Perchloroethylene
PDFID	Preconcentration Direct Flame Ionization Detection
PE	Performance Evaluation Sample
PEL	Permissible exposure limit
PHC	Petroleum Hydrocarbons (see TPH)
PID	Photoionization Detector
PM	Program Manager
PM_{10}	Particles having an aerodynamic diameter less than or equal to 10 micrometers
POC	Point of Contact or Point of Compliance
POHC	Principle Organic Hazardous Compounds
POTW	Publicly Owned (sewage) Treatment Works
POX	Purgeable Organic Halogens
ppb (PPB)	Parts per billion, micrograms per liter (µg/L), or micrograms per kilogram (µg/kg)
PPE	Personal Protective Equipment
ppm (PPM)	Parts per million, milligrams per liter (mg/L), or milligrams per kilogram (mg/kg) (in air, by volume)
ppt (PPT)	Parts per trillion, nanograms per liter (ng/L), or nanogram per kilogram (ng/kg)
PQL	Practical Quantitation Limit
PRP	Potentially responsible party held by EPA to be responsible for the remediation of a contaminated site under CERCLA
psi	Pounds per square inch
psig	Pounds per square inch gauge pressure
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene (e.g. Teflon®)
PUF	Open celled polyurethane foam used as an air collection media for semivolatile organic chemicals such as polychlorinated dioxins

PVC	Polyvinyl Chloride
PWS	Public Water System

Q

QA/QC	Quality Assurance/Quality Control
QAP	Quality Assurance Plan

R

RCRA	Resource Conservation & Recovery Act (1976)
RD	Remedial Design
RI/FS	Remedial Investigation/Feasibility Study
ROD	Record of Decision
RPPM	Respiratory Protection Program Manager
RQ	Reportable Quantity

S

SAP	Sampling and Analysis Plan
SAR	Supplied Air Respirator
SARA	Superfund Amendments and Reauthorization Act
SAS	Special Analytical Services (part of the EPA contract laboratory program)
SASS	Source Assessment Sampling System
SCBA	Self Contained Breathing Apparatus
SCS	Soil Conservation Service
SDWA	Safe Drinking Water Act (1974)
SIP	State Implementation Plan
SOC	Synthetic Organic Chemicals
SOP	Standard Operating Procedures
SOW	Scope of Work or Statement of Work
SPCC	Spill Prevention Control and Countermeasure Plan
SUMMA®	Registered trademark of Melectrics Corporation for a polishing and passivating for the interiors of stainless steel canisters used in air sampling

SVOC	Semivolatile Organic Compounds
SWDA	Solid Waste Disposal Act
SWMU	Solid Waste Management Unit (RCRA)

T

TAL	Target Analyte List (Inorganics - defined in CLP protocols)
TAT	Turnaround Time
TCDD	Tetrachlorodibenzodioxin
TCDF	Tetrachlorodibenzofuran
TCL	Target Compound List (Organics - defined in CLP protocols)
TCLP	Toxicity Characteristic Leaching Procedure
TCR	Total Coliform Rule
TDS	Total Dissolved Solids
TEGD	Technical Enforcement Guidance Document (EPA, 1986)
THM	Trihalomethane
TIC	Tentatively Identified Compounds or Total Ion Chromatogram
TIP	Transportation Improvement Program
TNCWS	Transient Non-community Water Systems
TOC	Total Organic Carbon
TOX	Total Organic Halogen Analysis
TPH	Total Petroleum Hydrocarbons (see PHC)
TSCA	Toxic Substances Control Act
TSD	Treatment, storage and disposal facility as defined under RCRA
TSD(F)	Treatment, Storage or Disposal (Facility)
TSP	Total Suspended Particulate
TSS	Total Suspended Solids

U

UEL	Upper Explosive Limit
UFL	Upper Flammable Limit

UL	Underwriters Laboratory
USCG	United States Coast Guard
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UST (UGST)	Underground Storage Tanks
UV	Ultraviolet

V - Z

VOA	Volatile Organic Analyte
VOC	Volatile Organic Compounds
VOST	Volatile Organic Sampling Train
WP	Work Plan
WQC	Water Quality Criteria
XAD-2	See Amberlite®

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