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## **Environmental Sampling Guide**

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DEPARTMENT OF THE ARMY  
U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY  
ABERDEEN PROVING GROUND, MARYLAND 21010-5422



REPLY TO  
ATTENTION OF

HSHB-ME-S

February 1993"

USAEHA TECHNICAL GUIDE NO. 155  
ENVIRONMENTAL SAMPLING GUIDE

CHAPTER 1  
INTRODUCTION

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**1-1. PURPOSE**

This standardized technical guide (TG) provides quality control (QC) guidelines for environmental sampling. Its purpose is to provide guidance to U.S. Army Environmental Hygiene Agency (USAEHA) personnel on how to sample water, soil, wastes, and other materials.

**1-2. REFERENCES**

Publications and forms are listed in Appendix A.

**1-3. ABBREVIATIONS, TERMS, AND TRADEMARKED NAMES**

Abbreviations, terms, and trademarked names used in this TG are explained in the glossary.

**1-4. BACKGROUND**

a. The USAEHA Regulation 702-I--

(1) Sets forth responsibilities for quality assurance (QA)/QC regarding environmental and occupational health sampling and analysis.

(2) Assigns primary QC responsibilities within a given technical area to the respective USAEHA program manager.

b. Historically, USAEHA program managers have developed their own internal guidance documents for environmental sampling of the particular media/parameters of interest to their program.

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\* This technical guide supersedes USAEHA TG No. 155, May 1991

c. We recognize that environmental Sampling requirements (both in media and analytes) often overlap program areas. Therefore, respective program managers and personnel from the Directorate of Laboratory Services (DLS) agree to standardize environmental sampling methods and QC requirements.

d. This TG--

(1) Should reduce the confusion about sampling by simplifying as well as standardizing the procedures used throughout USAEHA.

(2) Will serve as a training and guidance document for new project officers.

(3) Will serve as a reference and review document for the more experienced project officers.

(4) Will provide documentation to U.S. Environmental Protection Agency (EPA) or State regulators as to USAEHA's sampling methods and protocols.

e. The overall impact will be more consistent and meaningful results for support projects and special services performed by USAEHA.

#### 1-5. USER STATEMENT

This TG does not provide guidance on when, where, or why to sample but only on how to sample. It remains the individual program manager's responsibility to develop the guidance/rationale on when, where, or why to sample.

#### 1-6. TECHNICAL ASSISTANCE

Table I-I provides a list of program numbers with corresponding divisions and extensions. You should contact the appropriate program manager for technical assistance.

Table f-1  
Program Number Identification

| Program Number | Program Identification                  | Division                                 | Telephone* Extension |
|----------------|---|--|----------------------|
| 17             | Pesticide Monitoring                    | Pest Management and Pesticide Monitoring | 4131                 |
| 27             | Industrial/Environmental Health Physics | Health Physics                           | 3502                 |
| 28             | Medical Health Physics                  | Health Physics                           | 3502                 |
| 31             | Water Supply Management                 | Water Quality Engineering                | 3919                 |
| 32             | Wastewater Surveillance and Management  | Water Quality Engineering                | 3816                 |
| 37             | Hazardous Waste and Medical Waste       | Waste Disposal Engineering               | 3651                 |
| 38             | Ground Water and Solid Waste            | Waste Disposal Engineering               | 2024                 |
| 39             | Health Risk Assessment                  | Waste Disposal Engineering               | 2024                 |
| 74             | Quality Assurance                       | Analytical Quality Assurance             | 3269                 |
| 76             | Organic Analyses                        | Organic Environmental Chemistry          | 2208                 |
| 78             | Inorganic and Radiological Analyses     | Radiological and Inorganic Chemistry     | 2637                 |

\* If you are calling - DSN, dial 584-Extension  
 - Commercial, dial 30 I-67 1 -Extension

**CHAPTER 2  
APPROACHES TO THE SAMPLING PROJECT**

---

**2-1. INTRODUCTION**

a, Sites will vary in nature and complexity, and sampling projects must reflect the specific needs of each site. While there are many sampling methods and approaches, the project officer must develop a sampling plan that--

- (1) Considers methodologies consistent with the views and policies of regulatory agencies.
- (2) Balances organizational factors such as cost, time, and manpower.

b. The end result of any sampling effort should be the acquisition of representative samples that will yield valid data. The project officer can then determine the nature and extent of any contamination at the study site.

c. Several documents have been published by EPA which address the issue of sampling soils, sludges, liquids, and hazardous waste (see Appendix A). The project officer--

- (1) Should be familiar with these documents before developing and implementing a sampling plan.
- (2) Must follow related guidance/requirements provided by the State or local regulatory authority.

These documents contain site investigation methodologies approved by EPA for use by the regulated community. All sampling must be done in accordance with these references unless specifically directed to do otherwise by the State or local regulatory authority.

d. The USAEHA has had considerable experience with conducting site investigations, to include extensive sampling and analysis. While Department of the Army (DA) installations have many operations in common with private industry, there are several operations that are unique to the Army. The organizational knowledge for sampling studies can be found in experienced field personnel and Agency reports. These sources of information should be consulted when conducting an investigation.

**2-2. SAMPLING PLAN**

a. Developing a sampling plan is the first step in performing a sampling project.

(1) In some cases, the EPA or State regulator may require a plan to be submitted and approved before sampling may begin.

(2) In every case, however, the sampling plan is essential to a successful project whether or not it is formally written.

b. The sampling plan attempts to define what strategies and methods will best fulfill the objectives of the investigation. Figures 2-1 and 2-2 show an outline of the areas to be addressed in the development of a sampling plan. (Major areas will be discussed in more detail later in this chapter.)

### 2-3. SAMPLING SCHEME

Central to the development of a sampling plan is the sampling scheme. Also referred to as the sampling design, it includes sample type, sampling strategy, and number of samples.

#### a. Sample types.

(1) **Grab samples.** Also called single samples, Grab samples are discrete representations of conditions at a specific location and time.

(2) **Composite samples.** Composites are nondiscrete samples consisting of more than one specific aliquot collected at various locations and/or times. The analytical result represents average value but no measure of the precision of the value is obtained. Examples of composite samples are--

(a) Time and flow composites (often used in wastewater sampling).

(b) Depth composites (often used in soil sampling during drilling projects).

(c) Area composites (often used when sampling over large areas)

Specific uses of these types of samples are addressed in individual chapters of this TG.

(3) **Control samples.** While this type of sample is an important part of any sampling effort, controls are very important in projects involving land or water contamination caused by a release of hazardous constituents.

(a) An ideal control sample is a sample that is unaffected by the conditions being studied.

(b) A typical control sample is a grab or composite sample taken upgradient from the site suspected of being contaminated.

(c) The purpose of a control sample is to verify that the site in question is at the source of contamination.

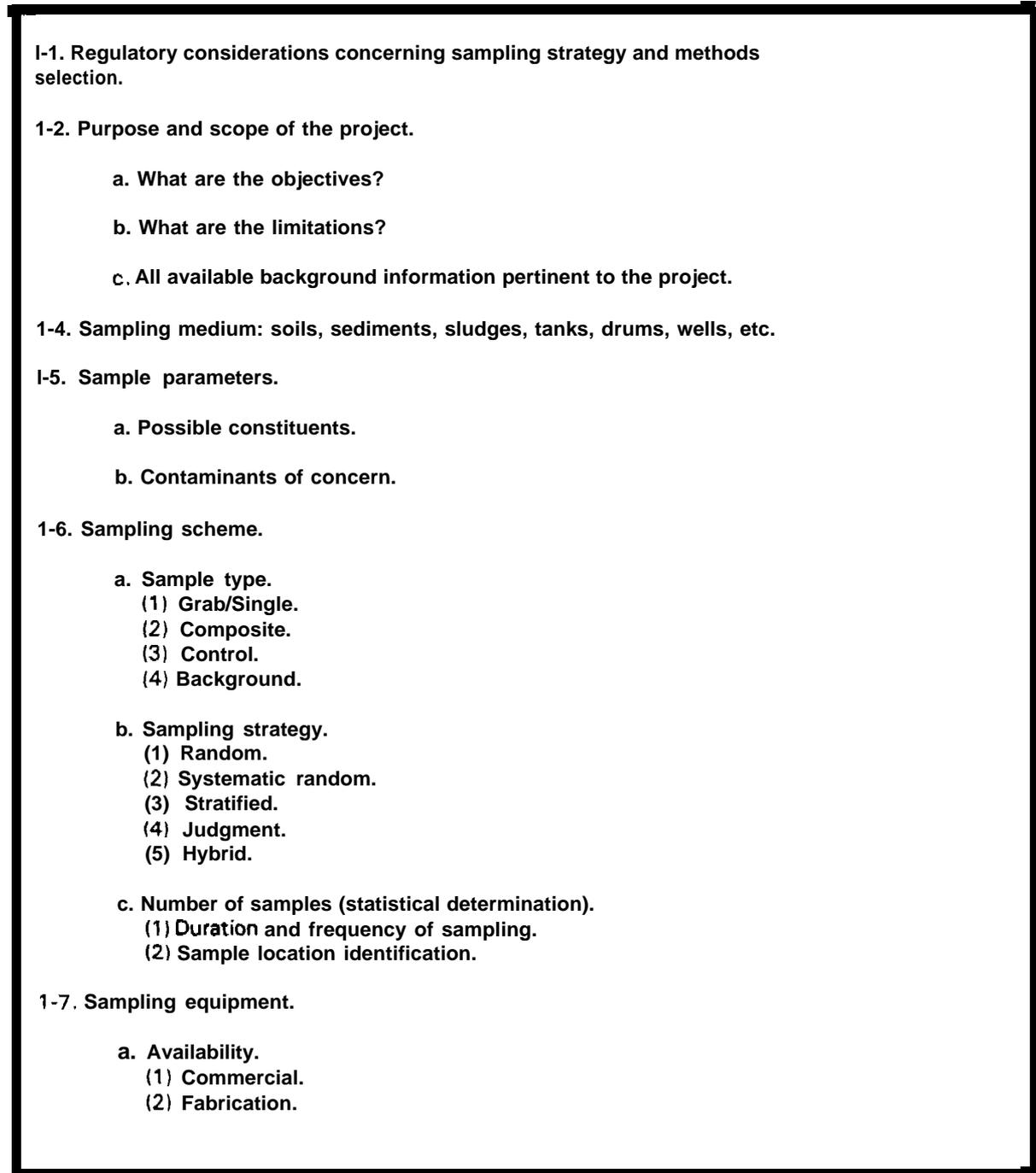


Figure 2-1. Sample Outline for Developing a Sampling Plan

- b. Suitability.
  - (1) Reusable.
  - (2) Disposable.
  - (3) Compatible.
  - (4) Contamination free.
- 1-8. Field methodology
  - a. Cleaning procedures for sample containers.
  - b. Decontamination procedures for sampling equipment.
  - c. Sample preparation in the field (including preservation information).
  - d. Sample containers.
  - e. Sample labeling and handling procedures.
  - f. Safety procedures.
- 1-9. QA/QC considerations.
  - a. Chain-of-custody.
  - b. Documentation and recordkeeping.
  - c. Calibration of field and laboratory instruments.
  - d. Preparation of QC samples.
- I-1 0. Time schedule.
  - a. Completion of sampling.
  - b. Sample analysis.
  - c. Report of results.
- I-1 1 . Sample analysis.
  - a. Analytical methods.
  - b. Detection limits.

Figure 2-2. Sample Outline for Developing a Sampling Plan (Continued)

(d) A control sample, which shows comparable levels of contamination, may indicate that--

1. The suspected site is not, in fact, the actual source of contamination.
2. Gradients may be more complex than they appeared and should be reassessed.

(e) Examples of control samples are a--

1. Soil sample upgradient from a spill site.
2. Sample from a stream upgradient from a discharge point.
3. Ground-water sample from a monitoring well located upgradient from a sanitary landfill.

**(4) Background samples.** Background samples are often confused with control samples because they are also taken offsite. Like controls, backgrounds also may be composite or grab samples and are important to land or water contamination studies.

(a) The purpose of a background is slightly different however. This type of sample is used to indicate the various characteristics and constituents that are naturally present in the medium.

(b) Examples of background samples are from--

1. Water bodies near a surface water contamination point.
2. Soil types similar to the site in question.

(c) A background sample must never be taken in or downgradient from any site that is contaminated or is likely to have released contamination in the past.

(d) In some cases, one sample may serve as both the control and the background sample if it accomplishes the purposes of both.

**b. Sampling strategies.** The following are examples of strategies commonly used in soil sampling studies but are also applicable to other types of environmental sampling.

**(1) Random sampling.** Random sampling relies on theory to choose sampling locations (e.g., the use of a random number table to locate sampling points).

**(2) Systematic random sampling.**

(a) This strategy involves the random selection of an initial sampling point, with subsequent points chosen relative to that point in set increments. The end result is a grid-like pattern.

(b) This is a commonly used and effective strategy because it is based on nonbiased point selection and fully covers the area of investigation.

**(3) Stratified sampling.** This type of sampling involves the division of the sample population into groups based on knowledge of sample characteristics at these divisions (e.g., samples are collected at point of each change in physical or chemical properties).

**(4) Judgment sampling.**

(a) This type of sampling is based on the project officer's judgment according to a certain bias, without regard to randomization (e.g., a sampling project is based on the conclusions reached in a similar project).

(b) This strategy should not be used exclusively because it may produce poor quality data and improper conclusions.

**(5) Hybrid sampling.**

(a) This type of sampling uses a combination of strategies.

(b) Hybrid schemes are usually the method of choice as they can allow for greater diversity without compromising the objectives of the project.

**c. Number of samples.**

(1) Selecting the number of samples which satisfy the study objectives (i.e., data adequacy, regulatory concerns) while trying to minimize the costs for these samples is one of the most difficult tasks needed in developing a sampling plan.

(2) Commonly, the sample numbers are arbitrarily chosen based on past experience, professional judgement, etc. This approach often results in inadequate or insufficient data which does not fulfill the study objectives, and requires additional sampling. Statistical methods are available which allow the scientific selection of the number of samples required (Appendix A, references 20, 21, 23, 25, 26, and 42).

(a) These references should be consulted if such a statistical determination is desired.

(b) However, the equations used often rely on data from preliminary sampling results such as information which may not be available to the project officer. The project officer must then arrive at a somewhat subjective number and must be able to justify the decision in terms of the study objectives and the intended use of the analytical data.

**24. QUALITY ASSURANCE/QUALITY CONTROL**

**a. Objectives.** A successful QA/QC program is essential to every sampling study because it allows the evaluation and interpretation of meaningful data.

(1) An ideal QA program ensures quality data, and identifies and quantifies all sources of error associated with each step of the sampling and analytical effort.

(2) QC is the routine application of procedures used to maintain the desired standards of precision. These procedures are performed both in the field and in the laboratory.

**b. QA/QC plan.** Like the sampling plan, the preparation and execution of a QA/QC plan is of great importance to a sampling project.

(1) QA/QC considerations may be included in the sampling plan, or they may be in a separate plan.

(2) QA/QC measures must be integrated into every aspect of the sampling and analytical efforts to ensure that the quality of the data adequately meets the needs of the project.

(3) Specific needs or questions relating to QA/QC should always be discussed with the Analytical Quality Assurance Division (AQAD) and the appropriate laboratory prior to sampling.

(4) Figure 2-3 shows an outline of the areas to be addressed in the development of a QA/QC plan.

**c. QC samples.** Control and background samples [discussed in paragraphs 2-3a(3) and (4), respectively] may be classified as QC samples. They are used to provide comparisons or references in the sampling project. Other types of QC samples are used in studies to indicate and quantify the margin of error in the sampling and analytical effort. The EPA recommends (Appendix A, reference 42) that one of every 20 samples should be in each of the QC categories defined below:

**(1) Duplicate and split samples.**

(a) Duplicate samples are collected by taking two separate samples in the same location at the same time.

(b) Split samples are samples which have been divided into two or more containers from a single sample. Partitioning assumes adequate mixing because split samples must be homogeneous.

- I-1. The intended use of the analytical data and the precision and accuracy required for this use.
- 1-2. Field procedures which will ensure representative samples.
- 1-3. Preparation procedures to be followed for sample containers, field equipment, and laboratory instruments.
- 1-4. Field decontamination procedures which will minimize the chance of cross contamination.
- 1-5. Documentation of the sampling effort, including labeling of containers, logbook entries, and photographs (discussed in Chapter 3).
- I-6. Chain-of-custody procedures in the field, in transit, and in the laboratory (discussed in Chapter 3).
- I-7. Sample handling guidance to include preservation, holding times, containers, and volume.
- I-8. Number and types of QC samples to be prepared in the field.
- 1-9. Number and types of QC samples to be prepared in the laboratory.
- I-I 0. Procedures for the documentation and calibration of laboratory and field instruments to acceptable limits specified by EPA or the manufacturer.
- I-1 1. Analytical data reviewing and reporting procedures.

Figure 2-3. Sample Outline for Developing a Quality Assurance/Quality Control Plan

(c) Duplicates and splits should always be carefully documented in the sampling plan and field logbook. They are used to--

1. Provide identical samples to opposing sides in a litigation situation.
2. Estimate sampling variability.
3. Provide material for matrix spiking when determining recovery rates.
4. Estimate recovery rate for the sample extractions.
5. Fulfill a requirement of regulatory agencies.
6. Serve as a QC check on the laboratory performing the analysis.

(2) Spiked samples.

(a) Spiked samples are prepared by adding a known amount of reference chemical to one of a pair of split samples. Results--

1. Are used to compute recovery efficiencies for analytes.
2. Elucidate interactions. That is, they show how the measurement for one compound may be affected by the presence or absence of another compound.

(b) Spiked samples are difficult to prepare in the field; therefore, we recommend that spiking be performed only as a laboratory procedure.

(c) In cases where field spikes are required by regulatory agencies, the appropriate laboratory must be consulted for assistance.

(3) Blank samples. Blanks provide a means of measuring the effect of various cross-contamination sources, background levels in the reagents, and decontamination efficiency. The types of blanks are--

- (a) Blank (an artificial sample designed to monitor artifacts in the process).
- (b) Reagent blank (an aliquot of analyte-free water or solvent analyzed with the analytical batch).
- (c) Field blank (an aliquot of analyte-free water or solvent brought to the field in sealed container). Two types of field blanks are--
  1. Trip blanks. These are not opened in the field. They check for sample contamination originating from sample transport, shipping, and from site conditions.
  2. Equipment blanks. These are opened in the field and poured over or through the sample collection device to detect contaminants on the device.

d. QC sample results. The QC results--

- (1) Should be included with the sample results when reported to the project officer.
- (2) Must be reviewed by an analyst at the laboratory and meet statistically valid control criteria, Sample data that fail to meet criteria must be identified.
- (3) Should also be discussed in the text of the project report.

**CHAPTER 3  
SAMPLE MANAGEMENT**

---

**3-1. INTRODUCTION**

**a. Proper sample management--**

- (1 ) Maintains sample integrity.
- (2) Ensures the safety of persons handling containers.
- (3) Enhances QC towards the production of valid data.

**b. This chapter deals with the general handling of samples and documentation of the sampling effort.**

**c. Specific techniques used in the collection of samples are addressed in the individual chapters devoted to each medium. The tables at the end of Chapters 4, 5, and 6 provide specific sample management information for parameters within the medium discussed.**

**d. Additional information for metals analyses is provided in Appendices B and C.**

**3-2. SAMPLE HANDLING**

**a. Sample containers.**

(1) In most cases, AQAD will supply the clean containers and labels for each project when advanced written notice is submitted 30 days prior to sampling.

(2) The project officer must specify the--

- (a) Medium or media to be sampled (for example, soil, water, etc.).
- (b) Analytes to be measured.
- (c) Number of samples expected.
- (d) Date the containers are needed.

(3) The project officer--

(a) May also request that containers be sent directly to the installation. We recommend that the project officer request a few extra containers to compensate for breakage or other circumstances requiring additional samples.

(b) Ensures that the correct types and numbers of containers are provided.

(4) Tables 4-1, 5-1, and 6-2 (located at the end of Chapters 4, 5, and 6, respectively) contain information required for different sampling media and analytes and required container types and sizes.

(5) If AQAD has not supplied new sample containers for a project, certain cleaning procedures are recommended to prepare containers for sampling. These procedures--

(a) Vary with the type of media sampled and analytes measured.

(b) Depend on regulatory requirements.

(6) Table 3-1, located at the end of this chapter, lists types of analyses and the container cleaning procedures recommended by the EPA (Appendix A, reference 48).

(7) Cleaning and decontamination procedures are also necessary for sampling devices and equipment. These procedures are--

(a) Outlined in each chapter devoted to a specific medium.

(b) Addressed in greater detail in paragraph 5-5.

#### b. Preservation.

(1) Chemical or physical preservation is required to prevent external contamination and maintain the integrity of some types of samples. Preservation techniques can only retard, not eliminate, the chemical and biological changes that take place in a sample after it has been collected.

(a) Chemical preservatives are added to the samples in the field and supplied to the project officer by AQAD along with the containers and labels.

(b) Other samples are physically preserved by cooling. As a general rule, we recommend that all samples be kept cool to minimize heat-induced reactions during transport.

(2) Preservation requirements for specific analyses in different media appear in Tables 4-1, 5-1, and 6-2.

(3) Samples that are improperly preserved or exceed maximum holding times will not be analyzed unless a strong valid justification is presented to the laboratory. Such data will be "flagged" to indicate increased uncertainty of values reported.

#### c. Holding times.

(1) Holding time is the elapsed time between sample collection in the field and analyte extraction in the laboratory.

(a) Certain holding times are mandated by law.

(b) Others are recommendations based on sample properties and the nature of the analysis.

(2) Analytical data that exceed maximum holding times must be so described and discussed in the project report. Such data are not legally valid. Every attempt should be made to have samples analyzed within the prescribed holding time.

(3) Project officers can assist with minimizing holding times by submitting samples as early as possible in the week.

(4) Tables 4-1, 5-1, and 6-2 list the maximum holding times for the various analytes in each medium. For analytes requiring very short holding times (48 hours), prior arrangements must be made with AQAD and the appropriate laboratory.

**d. Sample shipping.**

(1) Samples may be brought back from the field by the project officer or transported by a commercial carrier.

(a) The USAEHA frequently uses Federal Express or Express Mail to ship samples from the field to the laboratory. The appropriate forms are available through AQAD.

(b) Federal Express will not pick up samples on Sunday, and any samples sent on Saturday will be delivered on Monday.

(c) If samples are to be sent on Friday, the laboratory must be notified to ensure that a staff member will be present to receive the samples on Saturday. In some cases, the officer-of-the-day may receive the samples, but will not routinely transfer them to a permanent storage area, such as a refrigerator.

(d) Samples with a 48-hour maximum holding time, such as biochemical oxygen demand (BOD), require a chemist to work a weekend shift if samples arrive on a Friday. Project officers must give prior notification and communicate from the field if special scheduling of laboratory personnel will be required. The chemist must always have a way to contact the project officer, motel numbers are helpful if there is no other way to reach the project officer. If the scheduled to arrive on the weekend samples do not arrive the chemist must find out what went wrong and whether they should wait for the samples or not.

(2) Shipment of all types of samples must comply with Department of Transportation (DOT) regulations when applicable, as well as those of State and local governments.

(a) The person responsible for sample shipment (usually the project officer) must determine the appropriate shipping requirements, for example, proper labeling and packaging.

(b) Appendix D details the shipping requirements for environmental and hazardous samples.

(3) Suitable packing material should be used to prevent breakage of the samples.

(a) Include an absorbent in the shipping container to prevent leakage from a sample if it is damaged during shipment. This is especially important for samples containing polychlorinated biphenyls (PCBs), pesticides, or hazardous wastes.

(b) When shipping liquid samples, mark the level of the liquid in the sample container. This will aid laboratory personnel in locating a sample that has leaked during shipment.

(c) For samples that must be chilled during shipment, be sure to include an adequate supply of refrigerant to maintain the temperature for the duration of the transit period.

1. Prefrozen gel blocks are the best source to use. They cool more efficiently and remain cool longer than ice.

2. Do not use dry ice when shipping samples to the laboratory.

3. Do not let the refrigerant come in contact with sample containers because this can cause loss of labels and freezing of samples.

4. If ice must be used, it should be sealed in plastic bags.

(d) Table 3-2, located at the end of this chapter, describes refrigerant sufficient to achieve 3.5 - 5.5 °C.

(4) Small vessels containing liquids should be sealed in plastic bags or aluminum foil depending on the analysis requested (i.e., biological samples for pesticides measurements require aluminum foil; biological samples for metals require plastic bags). This prevents contamination of an entire shipment, if a sample leaks.

(5) When packing glass containers, wrap each container with insulation to prevent contact with other containers or the outer box. The samples should be packed to withstand a 6-foot drop.

### 3-3. DOCUMENTATION

#### a. General. Proper documentation--

(1) Provides a written record that ensures proper container identification.

(2) Maintains the security and integrity of samples.

(3) Is also useful to the project officer for interpretation of data and compilation of the final report.

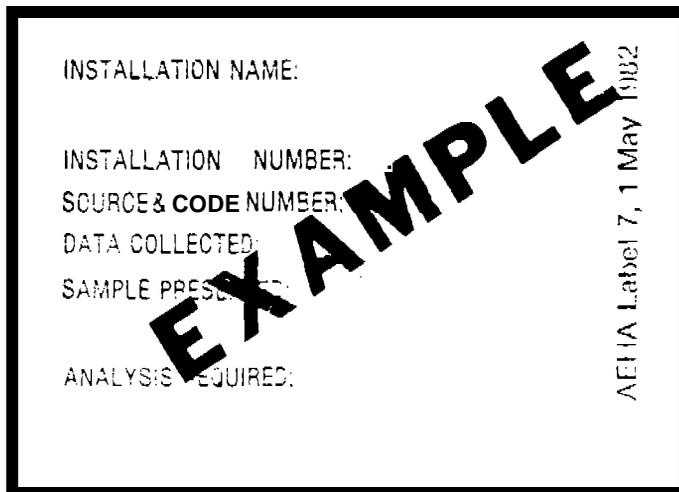
(4) Is an absolute necessity for projects that--

- (a) are highly visible or controversial,
- (b) are monitored by outside regulatory authorities, or
- (c) involve actual or potential litigation.

(5) Includes labeling containers, maintaining a chain-of-custody, keeping a field logbook, and submitting the proper analytical request paperwork.

**b. Container labeling.**

(1) All sample containers must be labeled with the USAEHA Label 7, shown in Figure 3-1, or an equivalent.



**Figure 3-1. Example of a Sample Container Label**

(2) Gummed labels or tags are acceptable. However, self-adhesive labels are less likely to fall off, especially if containers get wet.

(3) As a precaution, the sample number may be written on the container lid with waterproof ink to enable sample identification in case the label gets separated from the container.

(4) The AQAD will provide labeled sample containers, and each label will have the analytical requirements for the sample written on it.

(5) The project officer is responsible for adding project number, sample number, borehole number (when applicable), installation, collector's initials, project officer's name, and preservation method. The date and time (to the nearest hour) of collection must also appear on the label.

(6) The sample number must be written on the label at the time of collection to avoid misidentification. Waterproof ink is absolutely required for writing on sample labels.

c. Chain-of-custody. See Appendix E for a discussion of chain-of-custody policy.

d. Field logbook. All information pertinent to the investigation must be recorded in a field logbook. The book should be bound and numbered. Information recorded should be waterproof. This record is indispensable for data evaluation and report writing. Sufficient information should be recorded so that someone could reconstruct the sampling event without consulting the project officer. We recommend that the following information be recorded in the logbook:

- (1) Installation, project name, and project number.
- (2) Date and time of collection.
- (3) Any weather conditions which may have an affect on sampling.
- (4) Name(s) of sample collectors and assisting personnel.
- (5) A log of each sample collected, to include the--
  - (a) Type of sample.
  - (b) Place of collection.
  - (c) Analytes of interest.
  - (d) Sample number assigned.
- (6) Observations of the sample being collected (characteristics, odor, appearance).
- (7) Field measurements (pH, temperature, conductivity, etc.).

e. Requests for analytical support.

(1) Samples submitted to USAEHA for analysis must be accompanied with several completed forms describing the analyses requested and sample information (see Appendix F).

(2) In addition, a minimum 30-day advanced written notice must be submitted by memorandum to the appropriate laboratories and to AQAD.

(3) Appendix F details the requirements for submitting samples to USAEHA. Additional direct coordination with the laboratories concerning special projects or specific questions is strongly recommended.

**Table 3-1**  
**Superfund and Non-Superfund Container Cleaning**

**A. Superfund Containers**

| <u>Container Type</u> | <u>Container Size</u>            | <u>Sample Type</u>  |
|-----------------------|----------------------------------|---|
| A                     | 80-ounce (oz) amber glass bottle | Extractable organics  |
| E                     | 16-oz wide-mouth glass jar       | Extractable organics<br>Metals in soils and medium/<br>high concentration water |
| F                     | 8-oz wide-mouth glass jar        | Extractable organics<br>Metals in soils and medium/<br>high concentration water |
| G                     | 4-oz wide-mouth glass jar        | Extractable organics<br>Metals in soils and medium/<br>high concentration water |
| H                     | 1-liter (L) amber glass bottle   | Extractable organics  |
| J                     | 32-oz wide-mouth glass jar       | Extractable organics<br>Metals in soils and medium/<br>high concentration water |
| K                     | 4-L amber glass bottle           | Extractable organics  |

**Cleaning Procedure:**

- . Wash glass bottles, Teflon' liners, and caps in hot tap water with laboratory grade nonphosphate detergent.
- . Rinse three times with tap water.
- . Rinse with a 1: 1 mixture of nitric acid (HNO<sub>3</sub>) [metals-grade HNO<sub>3</sub> and American Society for Testing and Materials (ASTM) Type I deionized water].

**Table 3-1**  
**Superfund and Non-Superfund Container Cleaning (Continued)**

- Rinse three times with ASTM Type I organic-free water.
- Oven dry at 125 °C for 1 hour.
- Rinse with pesticide grade hexane using: 20 milliliter (mL) for ½-gallon (gal) container; 10 mL for 32-oz and 16-oz containers; and 5 mL for 8-oz and 4-oz containers.
- Oven dry at 125 °C for 1 hour. Allow to cool to room temperature in an enclosed contaminant-free environment.

Place liners in lids and cap containers.

Label each container with lot number and pack in case.

Label exterior of each case with lot number.

Store in contaminant-free area. (Amber glass containers shall not be exposed to sunlight.)

| <u>Container Type</u> | <u>Container Size</u> | <u>Sample Type</u>            |
|-----------------------|-----------------------|-------------------------------|
| B                     | 40-mL glass vial      | Purgeable (volatile) organics |
| D                     | 120-mL glass vial     | Purgeable (volatile) organics |

**Cleaning Procedure:**

- Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot tap water using laboratory grade nonphosphate detergent.
- Rinse three times with tap water.
- Rinse three times with ASTM Type I organic-free water.
- Oven dry vials, caps, septa, and liners at 105 °C for 1 hour

**Table 3-1**  
**Superfund and Non-Superfund Container Cleaning (Continued)**

- Allow vials, caps, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.
- Seal 40-mL vials with septa (Teflon side down) and cap.
- Place liners in lids and cap 120-mL vials.
- Label each vial with lot number and pack in case.
- Label exterior of each case with lot number.
- Store in contaminant-free area.

| <u>Container Type</u> | <u>Container Size</u>       | <u>Sample Type</u>       |
|-----------------------|-----------------------------|--------------------------|
| C                     | 1 -L polyethylene bottle    | Metals, cyanide, sulfide |
| L                     | 1 00-mL polyethylene bottle | Metals, cyanide, sulfide |

**Cleaning Procedure:**

- Wash polyethylene bottles and caps in hot tap water with laboratory-grade nonphosphate detergent.
- Rinse with 1 :1 HNO<sub>3</sub> mixture (metals-grade HNO<sub>3</sub> and ASTM Type I deionized water).
- Rinse three times with ASTM Type I deionized water.
- Invert and air dry in contaminant-free environment.
- Cap bottles.
- Label each container with lot number and pack in case.
- Label exterior of each case with lot number.
- Store in contaminant-free area.

Table 3-1  
 Superfund and Non-Superfund Container Cleaning (Continued)

**B. Non-superfund Containers**

| <u>Container Type</u> | <u>Container Size</u>            |
|-----------------------|----------------------------------|
| M                     | 1 -gal polyethylene cubitainer   |
| N                     | 2-L amber polyethylene bottle    |
| P                     | 500-mL amber polyethylene bottle |

**Cleaning Procedure:**

- Rinse containers three times with deionized water.
- Let stand for 48 hours with deionized water.
- Dry each container and place in clean plastic bags.

| <u>Container Type</u> | <u>Container Size</u>      |
|-----------------------|----------------------------|
| S                     | 125-mL polyethylene bottle |

**Cleaning Procedure:**

- Rinse containers three times with deionized water.
- Rinse containers three times with 3 normal (3N) HNO<sub>3</sub>.
- Rinse containers six times with deionized water.
- Let stand for 48 hours with deionized water.
- Dry each container and place in clean plastic bags.

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Table 3-1  
 Superfund and Non-Superfund Container Cleaning (Continued)

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| <u>Container Type</u> | <u>Container Size</u>              |
|-----------------------|------------------------------------|
| T                     | 50-mL polyethylene centrifuge tube |
| V                     | 15-mL polyethylene centrifuge tube |

**Cleaning Procedure:**

- Rinse containers three times with deionized water.
- Rinse containers three times with 3N HNO<sub>3</sub>.
- Rinse containers six times with deionized water.
- Let stand for 48 hours with deionized water.
- Empty containers and air dry in a LAMINAR<sup>1</sup>-flow hood delivering class 100 air,
- Place containers in clean plastic bags.

| <u>Container Type</u> | <u>Container Size</u>            |
|-----------------------|----------------------------------|
| R                     | 250-mL amber polyethylene bottle |

**Cleaning Procedure:**

- 50 percent of Type R containers are to be cleaned by the procedures used for containers Type M, N, and P. To distinguish that container Type R has been acid-washed, a round orange sticker shall be applied to the side of the container.
  - The remaining 50 percent Type R containers are to be cleaned by the procedures used for container Type S.
-

**Table 3-2**  
**Refrigerant Requirements**

| Container Size       | Pounds of Prefrozen Blocks | Hours Maintained Between 3.5 °C - 5.5 °C |
|----------------------|----------------------------|--|
| Small [½ pint (pt)]  | 3.5                        | 111                                      |
| Medium (1 pt)        | 7.5                        | 116                                      |
| Large [1 quart (qt)] | 18.0                       | 122                                      |
| Extra Large (1 gal)  | 21.0                       | 120                                      |

NOTE: All containers should be precooled to 4.0 °C before packing

CHAPTER 4  
COLLECTION OF WATER SAMPLES

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SECTION I - WASTEWATER

**4-1. INTRODUCTION**

Generally, wastewater sampling studies can be categorized as: permit or regulatory sampling of outfalls, sampling to determine unit process efficiency, and sampling within a collection system.

**a. Permit/Regulatory.**

(1) Installations with new treatment facilities, new point sources, or storm water discharges may be required to apply for a National Pollutant Discharge Elimination System (NPDES) permit or State equivalent (or be subject to pretreatment standards).

(2) Applications generally require analytical information as specified in parts 122, 124, and 125, title 40, Code of Federal Regulations (CFR) (40 CFR 122, 124, and 125). Some industrial operations are required to perform extensive quantitative analyses, including cyanide, phenols, metals, and various fractions of priority pollutants. We can assist in these one-time application efforts.

(3) Other studies will look at compliance with an existing permit (NPDES or pretreatment). Usually, regulatory-related studies are required to use analytical methodology specified by the NPDES program (40 CFR 136). This is an important point since USAEHA laboratories can use different methodologies for a given parameter, each with a different detection limit and difficulty of accomplishment. USAEHA must be informed of the regulatory nature of the sampling to ensure that correct procedures are used.

**b. Treatment system evaluations.**

(1) Many wastewater sampling studies are aimed at determining the efficiency of a unit process or group of unit processes.

(2) The number and types of analyses will differ according to the given treatment unit and the objective of the study.

**c. Collection systems.**

(1) Studies are conducted to locate the source of particular pollutants entering the wastewater (or storm water) collection system or to characterize various waste streams.

(2) Sampling takes place upstream of the treatment facility out of manholes, holding basins, troughs, or other conveyance/holding system.

(3) Characterization studies often include extensive flow monitoring in conjunction with sampling.

#### 4-2. SAMPLING EQUIPMENT

a. Automatic samplers are extremely useful when sampling wastewaters. They add a great deal more flexibility to studies since they can be programmed to sample at predetermined times and to collect predetermined volumes without constant supervision.

b. USAEHA has two basic models of automatic samplers: the ISCO<sup>®</sup> 2100 and ISCO 2700 models.

(1) Each sampler can be--

(a) Used with composite (single container) bases or discrete (multi-container) bases,

(b) Connected to a flow meter to collect composite samples.

(c) Powered by 12-volt direct current (DC) batteries, thus allowing sampling in remote areas.

(2) The ISCO 2700 model is more state-of-the-art and preferred when available.

(3) Both models use a peristaltic pump with silicon tubing. Silicon (general use) or Teflon (high purity use, such as for priority pollutant sampling) tubing is used between the wastewater and the sampler pump casing.

NOTE: The pumps in the different models operate at different intake velocities; thus when total suspended solids (TSS) analyses are important, use the same model sampler throughout.

c. Several types of grab sampling equipment are available, primarily for sampling sludges. Specialized grab samplers can be fabricated in many cases to handle unique situations. Additionally, the Water Quality Engineering Division (WQED) maintains a wide variety of liquid monitoring devices (such as pH recorders, conductivity meters, etc.) to augment sampling efforts.

d. The types of equipment to be used at a particular site should be worked out between the project officer and the project engineering technician.

#### 4-3. CLEANING PROCEDURES

a. General.

(1) In general, sample containers will be provided by AQAD. One-gallon amber jugs are maintained in the WQED warehouse. Thus, for most surveys cleaning of containers is not necessary.

(2) When using an automatic sampler in the field, new tubing is always installed at the beginning of the survey, but is not normally replaced daily. This is acceptable if the sampler will be used at only one location. If it is necessary to move the sampler to a different location, install new tubing.

(3) When sampling for priority pollutants or for regulatory purposes, more specific and stringent cleaning procedures are required.

b. **Containers.** Most containers should be already cleaned by AQAD prior to the survey. If not, use the cleaning method as specified for container types A through H, J, and K in Table 3-1.

c. **Equipment.** It is often necessary to clean sampling equipment in the field to minimize the potential of cross contamination between sample points. This is particularly essential when priority pollutants are being analyzed. Taking extra equipment may alleviate the problem. However, this is not always practical. The following procedures should be used to clean equipment onsite:

- (1) Wash with detergent.
- (2) Rinse with tap water.
- (3) Rinse with pesticide grade acetone.
- (4) Rinse with deionized water.
- (5) Air dry.
- (6) Rinse with pesticide grade petroleum ether.
- (7) Air dry.
- (8) Wrap with aluminum foil to protect it to the next sample point.

**NOTE:** Step (6) may be omitted if priority pollutants are not a concern. The use of equipment blanks should also be considered to improve the quality of data.

#### 4-4. SAMPLE COLLECTION

##### a. General.

(1) The project officer, upon detailed study of the given situation (usually via a preliminary site visit) and in conjunction with engineering technician consultation, determines--

- (a) Representative locations.
- (b) Types of equipment to be used.
- (c) Number and types of samples to be collected.

(2) A study protocol should be established for most studies with input from laboratory and installation personnel. Peer input can also be helpful. A good protocol can help eliminate potential problems.

(3) The project officer must be sure to collect the proper volumes for the type of sample and specific parameters of interest. See Appendix F for additional information on laboratory coordination. The two general types of samples are grab samples and composite samples, which can be further divided into time- and flow-composite types.

**b. Sample volume.**

(1) Required sample volumes for individual parameters are listed in Table 4-1, which is located at the end of this chapter. It is always wise, however, to check with individual laboratories concerning the volume required.

(2) Sometimes additional samples may be requested or required. The AQAD will supply containers and labels for requested parameters.

(3) Some parameters which require the same preservative are often combined into a single container. For example, nitrate/nitrite-nitrogen, ammonia-nitrogen, and total Kjeldahl nitrogen (TKN) are often collected in a single 1-qt cubitainer.

(4) When composite sampling, the individually required volumes must be summed. This sum will give the minimum amount to be composited. As a safety measure, multiply this amount by 1.5 to determine the actual amount to collect.

**c. Grab sampling.**

(1) Always check with the appropriate laboratory for specific parameters.

(2) A grab sample is required for certain tests that must be performed as soon as (or soon after) the sample is collected: pH, chlorine residual, dissolved oxygen, temperature, and fecal coliform (FC).

(3) Samples for grease and oil, volatile organic compounds (VOCs), cyanide, and total phenols are collected as grab samples (see Table 4-1), because automatic samplers are inappropriate (e.g., oily materials will adhere to silicon tubing).

(4) Other situations when a grab sample may be used are when--

(a) The waste to be sampled is fairly consistent and does not require a composite sample (e.g., sludges, some lagoons, and surface waters).

(b) An attempt is being made to isolate a "dump" into a collection system.

(c) The wastewater stream is intermittent.

(d) The NPDES permit requires a grab sample.

(e) The location prohibits collection of composite samples.

d. Composite sampling.

(1) A composite sample is a number of Grab samples collected over a period of time (most often, 24 hours) and combined. An automatic sampler will combine grabs into a single container.

(2) The NPDES program requires at least 8 aliquots (grabs) of a minimum 100 mL each over a 24-hour period. The more often a sample is collected, the better the composited sample will reflect actual conditions. However, while an automatic sampler can take up to a sample per minute, the total composite (container within sampler) is normally limited to 5 gal (18.75 L).

(3) Typically, samples are collected at intervals of between 15 minutes and 1 hour. The total amount of sample collected is based on the analytical requirements plus a safety factor.

(4) The composite sample may either be a time-composited sample, in which the time and grab sample size remain constant, or a flow-composited sample, in which the sample size remains constant, but the time interval varies according to the flow passing the sample location. Flow-composited samples will normally be more representative of the waste stream, but require use of a flow metering device or use of flow data obtained elsewhere (such as from an upstream meter or from fixed treatment plant recorders).

NOTE: When many sample points are composited, the collection of samples will result in many composite containers being moved to the breakdown area. Label or mark the composite containers by sample point to avoid confusion.

(a) Time-composited sample.

1. Manually collected or sampler collected grabs taken over constant time intervals may be used when--

a. The flow rate does not vary more than 15 percent from average flow.

b. A unit process is evaluated that does not have a long hydraulic detention time.

c. Flow monitoring is prohibitive due to lack of equipment or to lack of adequate monitoring location.

d. Quality objectives would not suffer. (There is a trade-off between data quality and the amount of time that must be devoted by the project team; time-composited samples take much less time for the team than flow composites.)

2. There are four key variables in time-composited sampling--

- 2. Time interval between samples.
- b. Volume of individual grabs.
- c. The number of samples to collect.
- d. The total composite volume to collect.

(1) Normally, the-total composite volume to collect is known (amount needed for analyses plus a safety volume).

(2) Once the composite volume is established, then either the time between samples or the volume of each individual grab is arbitrarily chosen (fixing the time will also yield the number of samples to collect), although the permit may specify one or both of the variables.

(3) To calculate the volume of the individual samples using a pre-chosen time interval:

$$\frac{\text{Final Sample Volume}}{\text{(Composite Period/Time Interval)}} = \text{Fixed Volume per Sample (mL)} \quad (1)$$

01

$$\frac{\text{Final Sample Volume}}{\text{Number of Samples}} = \text{Fixed Volume per Sample (mL)}$$

For example, assume that samples are to be collected every 30 minutes (over 24 hours) and that 12 L (12,000 mL) of sample are needed. Thus the amount to be sampled every 30 minutes equals 12,000/48, or 250 mL. The automatic sampler can be set at this pull. If this amount is too low (< 100 mL), then the time interval should be decreased accordingly. Alternatively, if the grab sample pull volume is preestablished, then the time interval between samples is calculated as follows:

$$\frac{\text{Final Sample Volume}}{\text{Fixed Volume}} = \text{Number of samples to collect} \quad (2)$$

and

$$\frac{\text{Sampling Period}}{\text{Number of samples to collect}} = \text{Time interval between samples} \quad (3)$$

g. Certain parameters, specifically grease and oil, cannot be composited using an automatic sampler. When a composite of grease and oil is desired, then manual grabs, collected over time, can be poured into a single container as a time composite.

(b) Flow-composite sample.

1. Flow meter tie-in.

a. In many cases, an automatic sampler can be connected to a flow monitoring device which sends a signal to the sampler each time a preset amount of flow passes by the device.

b. More samples will be drawn during high flow rates than at times when low flow rates exist. Because of this, flow-composited samples generally are more representative of actual wastewater conditions than time composited samples.

c. When using a flow meter in combination with a composite sampler, the calculations for determining a sample draw are straight-forward. However, a knowledge or good estimation of flow is a prerequisite.

(1) First, estimate a target number of samples to be taken. For example, a target of 50 samples is commonly used (about 2 per hour over 24 hours if flow conditions are steady).

(2) The equation is as follows:

$$\frac{\text{Average plant flow (gall)}}{\text{\# of samples to be collected}} = \frac{\text{interval flow setting}}{\text{on flow meter/sampler}} \quad (4)$$

(3) Assuming that at least 50 samples are to be collected and that the average daily flow by the point is 3 million gallons per day (MGD), then the interval flow setting would be 3 million gal/50 or 60,000 gal. One sample will be drawn after each 60,000 gal of flow passes the point. Program this setting into the sampler/flow meter setup.

(4) Then a volume per sample must be determined. If 10 L are required (based on analytical needs) then 10,000 mL/50, or 200 mL per pull is needed. Generally, some additional safety margin is included in case the daily flow is slightly different than expected. Check the composite volume using an expected range of flows. Caution: if the actual flow is far less than or far greater than the estimate, then the sample may be inadequate. Again, a good knowledge of expected conditions is essential.

(a) For example, at most Army bases weekend flow is significantly lower than weekday flow. In the example, if the Saturday flow is 1 MGD, then only 3.3 L of sample will be pulled. In this case the settings should have been changed to 20,000 gal intervals (or increase the individual sample pull or a combination of both).

(b) Conversely, if the collection system experiences high infiltration and inflow problems during rain events, then similar adjustments are necessary. For example, if the flow increased to 6 MGD due to heavy rains, then the theoretical amount of sample pulled would be 20 L. However, the sampler (ISCO 2700) would shut off when the composite container was full (about 15 L).

**2. Manual flow composite.**

**a.** A manually flow-composited sample consists of discrete sampling at timed intervals and use of flow data from another source (such as a flow meter on a unit process up or downstream of the sample point) or from a device that cannot be tied into the sampler (such as the treatment plant's flow meter).

(1) In these cases, samples are pulled at set time intervals into separate containers (up to a maximum 25 for the ISCO samplers).

(2) Then, proportionate amounts of each container are manually transferred to a single composite container, using the available flow data.

**b.** Manual compositing is time intensive, but will generally be more representative than a simple time composite. The choice will depend on the specific study.

**c.** In this mode, the sampler will draw water into individual bottles at preset time intervals. When using one sampler, each bottle will represent 1 hour of flow past the sample point (assuming a 24-hour composite). Generally, the sample is pulled at mid-hour. The sampler can also be set to put several samples into a single bottle, but bottle sizes must be taken into account:

(1) ISCO 2100 & 2700 plastic bottles - 1,000 mL capacity

(2) ISCO 2100 & 2700 glass bottles - 350 mL capacity

**d.** When samples are pulled following the compositing period, the flow record for the same period must also be pulled. The procedure for manual flow compositing follows:

(1) Analyze the flow record. Calculate the average flow for each hour recorded (or other unit time period, depending upon the compositing period chosen). Find the period(s) with the highest flow rate. This will be the 100-percent rate. As an example, assume the maximum hourly rate is 3 MGD.

(2) For the remaining periods, find the fraction of the peak flow. For example, the flow during one period averaged 2.5 MGD. The fraction is  $2.5/3$ , or 0.83.

(3) Multiply the fraction for each period by the amount collected in each bottle, say 1,000 mL using an ISCO 2100 with plastic bottles. This is the amount of sample to be transferred to the compositing container.

(4) For the above period, 830 mL would be transferred to the compositing container from the hourly sample. Note that the maximum amount of sample, using one sampler would be 24,000 mL and normally much less if peak flows differ substantially from low flows (as is often the case at Army installations). A good knowledge of the flow conditions is required.

(5) If the required sample volume is high, then two samplers can be used (one for the first half of the sampling period, the other for the second half). Use the same model sampler since the drawing power (pumping) of different models may differ (and may affect TSS and other results).

e. **Representative sampling.** Proper sampling efforts provide a representative sample of the waste stream for laboratory analysis. If the sampler uses sloppy or improper technique, or the samples are improperly preserved, then the data may be invalid. The following procedures should be followed to ensure a representative sample is taken:

(1) Sample each waste stream consistently in the same location. Thus the sample will be consistent and the test data can be compared from day to day.

(2) Rinse sampling devices thoroughly and keep strainers free of debris.

(3) Take samples where there is good mixing action in the waste stream. Waste streams are usually well mixed in an aerated channel. Samples should not be taken where--

(a) Solids deposit.

(b) There are large chunks of debris.

(c) Scum accumulates, such as in quiescent corners, in a channel, or at the surface of a clarifier (unless, of course, this material is necessary to the quality objectives of the survey).

(4) As with liquid samples--

(a) Sludge should be well mixed and should be taken at a location that is consistent and accessible.

(b) Sample bottles should have wide mouths to accommodate the sampling scoop or bottle. A space for gas collection should be left at the top of the bottle, and the cap should be left loose for gas to escape.

(c) For dewatered sludge, samples should be taken from several places to ensure a homogenous sample.

(5) An anaerobic digester gas is commonly sampled for methane, carbon dioxide, and hydrogen sulfide content. A sampling location must be chosen carefully, as air is a common contaminant. Digester gas often is sampled at the dome or in a pipeline to the waste gas burner or boiler.

f. **Weather conditions.** Generally, weather conditions will not hinder automatic sampling. However, there are a number of actions which may be necessary to avoid problems.

(1) During periods of warm weather, or when the sampler sits out in the sun for extended periods, it is wise to maintain 3 or 4 pounds of bagged ice in the center of the sampler, especially when biologically related parameters are to be analyzed (BOD). The ice will act to preserve the samples (retard bacterial action).

(2) During periods of below freezing weather, icing problems may develop. In this case, ensure that sample draw lines have no low spots where wastewater can lie between sample pulls. Heat tape can be wrapped around the tubing and the entire sampler can be placed under a protective structure with a light within. Keep sample lines as short as possible.

#### 4-5. SAMPLE MANAGEMENT

##### a. Containers.

(1) Sample containers will be provided by AQAD, as requested.

(a) Give AQAD enough advanced notice to prepare containers and then inspect containers for correct number and type before leaving for the survey.

(b) The AQAD can send sample containers to the installation ahead of the survey, but this requires additional advanced notice.

(2) Always take more containers than theoretically needed, to have enough in case of breakage or to take additional samples according to special onsite conditions.

(3) See paragraph 3-2a for additional information.

##### b. Preservation.

(1) Paragraph 3-2b contains general information concerning preservation. The AQAD will supply chemical preservatives.

(2) Table 4-1 lists the preservation requirements for most parameters in water. Check with the latest revision of 40 CFR 136 for current rules. Note that some parameters require no preservation other than cooling. Also, be aware of the holding times.

(3) Sludge and soil samples do not normally require preservation.

(4) Always allow some space at the top of the container for preservative addition.

(5) If a sample is taken for grease and oil and it overfills the container, do not pour out sample to make room for the preservative. Much of the floatable grease and oil material will be lost.

**c. Labeling**

- (1) Labels are normally supplied by AQAD in conjunction with the survey.
- (2) Blank labels are available from the WQED warehouse. Labels may also be created by using your computer and printer.
- (3) Labels must be clearly marked with date of collection, installation, and sample identification number.
- (4) Some labels may fall off during shipment. For that reason, some project officers number the sample point on the container lid so the laboratory can still possibly identify the container.
- (5) Numbering identification should be preestablished with the laboratories to avoid confusion when samples arrive at USAEHA.

**d. Sample shipping.** Samples collected in the field are normally sent back to the Agency through Federal Express. Federal Express forms are available through AQAD. See paragraph 3-2d for more details.

**NOTE:** If samples are sent on Friday, then someone in the laboratory will need to work on Saturday to receive the samples. Also, any 5-day BOD samples sent on Monday (Tuesday arrival) will require a chemist to work on the following Sunday to read final dissolved oxygen levels. In addition, samples with 48-hour holding times sent on Friday will require a chemist to work weekends. Weekend work must be coordinated with appropriate laboratories and should be noted when working out the analytical matrix in advance.

**4-6. SAFETY**

- a. There are many safety hazards which project personnel may encounter while sampling, such as: toxic gases, vapors, and fumes; toxic liquids; explosive conditions; slippery surfaces, moving equipment; etc. The topic is only mentioned here to stress its significance and will not be detailed.
- b. Appendix A, reference 47 is an excellent source on this topic. However, the project officer must assess the hazards on a site-by-site basis and ensure project personnel are cognizant of all hazards and also are protected against any type of potential hazard.

## SECTION II - GROUND-WATER MONITORING WELLS

### 4-7. INTRODUCTION

a. Ground-water monitoring wells are generally sampled in response to a regulatory requirement (Public Law (PL) 94-580, Resource Conservation and Recovery Act (RCRA) of 1976; or State) or in conjunction with a project. Parameters for analysis are chosen by the appropriate regulatory agency or by the project officer.

b. Although monitoring wells vary from site-to-site in size and construction materials, the procedures used to collect representative samples do not vary greatly. The process consists of--

- (1) Measuring the water level in the well.
- (2) Purging the well.
- (3) Collecting the samples.
- (4) Filtering and preserving the samples.

### 4-8. CHOOSING EQUIPMENT

#### a. General.

(1) Prior to proceeding with the purging and sampling process, the project officer must be familiar with the wells to be sampled and the parameters to be analyzed so that equipment can be chosen appropriately. Well diameter, depth to the water, total depth, accessibility, and estimated yields of the wells should be considered when choosing equipment for the purging and sampling procedures.

(2) Although choosing equipment for purging is not as critical as choosing equipment for sampling, it is usually more convenient to use the same item for both processes. The parameter list is probably the most important thing to consider when choosing the equipment.

(a) Some sampling devices involve contact between an introduced gas and the ground water, such as air-lift or air-pump samplers which use rising gas bubbles to reduce the average density of the water to lift it to the top. These are unacceptable because--

1. The mixing of air causes changes in the pH and oxidation state of the water which may result in the precipitation of metals and other contaminants.

2. Any VOCs would also be lost due to aeration.

(b) Jet pumps are adequate for evacuation of wells, but are unacceptable for sampling because foreign water is mixed with the ground water when obtaining a sample.

(3) The following bailers and pumps are generally available within USAEHA. Their use is recommended as discussed below.

**b. Bailers.**

(1) A bailer is a sampling tube which is lowered into a well, filled with water, and retrieved with a sample. Various sizes are available in different forms and materials.

(2) The main requirement is that the bailer be constructed of materials that will not react with the chemical parameters being analyzed. The recommended materials for a bailer are stainless steel and Teflon, and those with check valves (either single or double) are better than those without.

(3) Purging a well with a bailer is only recommended for low yield, small-diameter wells which can be bailed dry. A well which recharges too quickly to be bailed dry may never be bailed adequately enough to remove all of the standing water.

(4) The advantages of using a bailer include portability and easy cleaning, although a large amount of time and effort is required to bail wells.

(5) Teflon or stainless-steel bailers with check valves are acceptable sampling devices for collecting samples for VOCs.

**c. Suction samplers**

(1) Suction samplers include portable alternating current (AC) and DC peristaltic pumps and larger gasoline-powered centrifugal pumps. These are not "down-the-hole" types of pumps. They are above the surface and rely on the static water level being closer than 25 feet from the surface. The intake line extends down the well casing, evacuates the well through suction, and discharges the water through a discharge line on the surface. This type of pump can be used in 2-inch diameter and larger wells.

**(a) Peristaltic pumps.**

1. The advantages of a peristaltic pump are portability and low probability of cross contamination. Because the sample comes into contact with only the tubing, changes between wells will prevent cross contamination and eliminate the need to clean the equipment.

2. The main disadvantage of a peristaltic pump is the low pumping rate (1 L per minute) which makes it difficult and slow to purge a large volume from a well.

**(b) Larger gasoline-powered centrifugal pumps.**

1. This type of pump can be used if care is taken to pump large volumes of clean water through the hosing before purging and sampling.

2. The advantage of a centrifugal pump is the high pumping rate which makes it fast and easy to purge a large volume.

(2) Neither pump should be used to collect samples for VOCs.

d. Bladder pumps.

(1) Bladder pumps are excellent choices for 2-inch diameter wells because reliable purging and quality sampling can be conveniently accomplished.

(2) They are commonly constructed of stainless steel and Teflon, and operate with some type of gas, in most cases nitrogen.

(a) A flexible bladder inside a stainless-steel tube fills with water and empties at the surface when the pressurized gas (air or nitrogen) fills the space between the stainless-steel tube and the flexible bladder.

(b) Because there is no contact between the gas and the water, very little agitation of the sample occurs.

(3) Bladder pumps are acceptable and often the preferred method of sampling for VOCs.

(4) Most bladder pumps have regulators for adjusting the time interval between filling and emptying. The average pumping rate varies according to the depth of water in the well from near 0 to 1.5 gal per minute (gpm). Therefore, the time required to evacuate a small-diameter deep well may be substantial.

e. Submersible pumps. Submersible pumps are made in several different sizes suitable for 2-, 3-, and 4-inch diameter wells.

(1) All are acceptable for purging wells and sampling for most contaminants.

(2) The small-diameter submersible pumps are suitable primarily for use in 2-inch diameter wells.

(a) They are battery-driven, very portable, and can usually pump 0.1 to 1.5 gpm depending on the depth of water in the well.

(b) Pumping wells with fine sand and silt is not much of a problem because these pumps generally have a reverse switch to dislodge clogged material.

(c) Because a low-velocity, uniform, continuous flow is possible, samples for VOCs may be collected with these pumps if Teflon tubing is used,

(3) The large-diameter submersible pumps are suitable for use in 3- and 4-inch diameter wells.

(a) These are normally much heavier pumps, sometimes requiring tripods and pulleys to lower and raise them. A generator is needed to supply power.

(b) Their primary limitation is the tendency to jam when fine sand and silt is pumped.

(c) The pumping rate of 2 to 10 gpm is too high a velocity to collect good quality samples for VOCs.

**f. Construction materials.**

(1) The construction materials of the samplers are important in maintaining sample integrity.

(a) The best materials are stainless steel and Teflon, especially for sampling and analyzing for trace levels of heavy metals or organic compounds.

(b) Polyvinyl chloride (PVC) is cheaper and acceptable for most situations, but should be avoided when collecting for organic analyses.

(c) Bailers should be constructed of stainless steel and/or Teflon.

(2) The selection of tubing to use with a sampler will depend on the situation.

(a) Teflon is recommended for those sites where sampling and analysis for organic compounds is to be performed.

(b) Polyethylene and vinyl are acceptable in many other situations and are less expensive than Teflon.

(c) Neoprene' is less desirable and should never be used if the samples are to be analyzed for organics.

(d) Rubber is not recommended.

**4-9. WATER LEVEL MEASUREMENT**

a. Water level measurements must be made to determine the direction of ground-water flow and to calculate well volumes.

(1) The depth to the water surface is measured using either a chalked steel tape or an electrical water level indicator.

(2) All measurements should be made to the nearest 0.1 foot and to the same reference point (either the top of the casing or the ground surface) which must be surveyed for elevation.

(3) The water level elevation is calculated by subtracting the water level from the reference elevation (ground surface elevation or top of casing elevation).

b. All equipment used in measuring the water level should be washed in tap water and rinsed in distilled water before proceeding to the next well. If contamination by grease or oil is evident, soapy water or acetone should be used for cleaning, followed by tap water rinsing.

**4-1 0. WELL EVACUATION**

a. Because the standing water in a Well is not representative of the ground-water quality, a well must be purged prior to sampling.

(1) This can be accomplished by--

(a) Evacuating a minimum of three to five well volumes from the well, or

(b) Pumping water from the well until conductivity, pH, and temperature have stabilized.

(2) Low yield wells are often purged nearly dry.

b. One well volume can be calculated using the following formula:

$$R \times \frac{\text{total well depth}}{\text{in feet}} - \frac{\text{depth to the water surface}}{\text{in feet}} = \text{volume in gallons} \tag{5}$$

where R = 0.16 for 2-inch inside diameter (ID) wells

R = 0.37 for 3-inch ID wells

R = 0.65 for 4-inch ID wells

(1) The measurements for total well depth and depth to the water surface must relate to the same reference point (ground surface or top of casing).

(2) Multiply the value for 1 volume by 3 or by 5 to determine the minimum purge volume.

c. The volume purged from a well can be easily measured with an empty 5-gal bentonite bucket or a similar item.

d. Pumps or bailers may be used to purge ground-water monitoring wells as discussed in paragraph 4-8.

**4-1 1. CLEANING PROCEDURES**

a. All equipment including pumps, bailers, and filtering apparatus must be decontaminated between wells and before sampling the first well.

(1) **Pumps.** The standard cleaning procedures include pumping clean, potable water through the pumps and rinsing with distilled water:

(a) Normally, a clean barrel or trash can is filled with the water to be pumped, and the pump is placed in the barrel and pumped after each well.

(b) Distilled water is poured over the outside parts.

(2) **Bailers and filtering apparatus.** The cleaning procedures are similar to the ones used for cleaning pumps by using potable water and a distilled water rinse.

b. If grease and oil are suspected to be present in the well, the sampling device should be washed with soapy water, rinsed very thoroughly with potable water, and followed by distilled water rinse.

c. Acetone is an effective way to remove grease and oil but should not be used unless it is certain that the pump or bailer parts would not be affected.

d. One area frequently missed, in terms of cleaning, and a persistent source for cross contamination is the line attached to the sampling device. This line should be constructed of material which will not adsorb water from the well, and thereby, act as a trap or carrier for contaminants which can be transported to the next well.

(1) If a cloth line or similar material is used, the line should be dedicated to that particular well to minimize the potential for cross contamination.

(2) There are commercially available stainless-steel lines or Teflon-coated lines which can be easily cleaned in the field.

e. A sample of any chemical used to clean equipment should be collected and forwarded to the laboratory.

**4-12, SAMPLING PROCEDURES**

a. Samples should be collected immediately after purging is completed or, if the well was purged dry, as soon as the well recharges. In general, the upgradient well should be sampled first, and then sampling should proceed from the well suspected to be the least contaminated to the most contaminated.

b. The containers should be rinsed several times with a small amount of the well water before filling, except those samples for grease and oil. All containers except those for grease and oil should be filled, without excessive agitation, to overflowing to prevent Chemistry changes due to either carbon dioxide loss with pH increase or oxidation of the samples.

c. When sampling for VOCs, the 40-mL vials must be filled using the method described in paragraph 4-17c(4). For parameters which require filtering, fill gallon containers in the field.

#### 4-13. SAMPLE FILTERING

a. Table 4-1 lists the sample quantity required, preservation methodology, and filtration requirements. If filtration is required, the final product must be filtered through a 0.45-micron filter.

b. Those samples requiring filtration should be collected in 1-gal plastic jugs. Filtration must be accomplished as soon as possible after sampling, preferably within 2 hours. Although field filtration would be ideal, it is not often feasible. Therefore, filtration in a clean laboratory is acceptable.

c. Two types of methods are available for filtering ground-water samples.

(1) The vacuum method uses a vacuum pump to draw the water through the filter.

(2) The pressure method uses a pressurized inert gas (commonly nitrogen) to force the water through the filter. The pressure system is better than the vacuum system for two reasons.

(a) Quicker filtering can be accomplished with the pressure system. This is especially important if the samples contain large amounts of fine suspended material.

(b) Precipitation due to pH and oxidation changes is also less with a nitrogen pressure filtration system than with a vacuum system.

#### 4-14. SAMPLE MANAGEMENT

a. Normal procedures involving sample containers, preservation, holding times, and shipping are discussed in paragraph 3-2.

b. Container labeling, documenting, and Chain-of-Custody procedures are detailed in paragraph 3-3.

c. The appropriate sample volumes, containers, preservatives, holding times, etc., are included in Table 4-1 according to the analyses to be performed.

d. Requirements for the submission of samples to USA-EHA for analysis are detailed in Appendix F, and must be closely followed.

**SECTION III - POTABLE WATER****4-15. BACKGROUND**

Sampling of the potable water is generally performed in order to determine compliance with the National Primary Drinking Water Regulations (NPDWR) and the National Secondary Drinking Water Regulations (NSDWR). These regulations (40 CFR 141 and 143) were established to ensure safe drinking water would be provided to the consumer.

a. Under the NPDWR, maximum contaminant levels (MCLs) have been set for contaminants which could pose a direct health threat. Included are inorganic, organic, radiochemical, and microbiological contaminants. Many more contaminants are scheduled for regulation over the next few years.

b. The NSDWR are Federally unenforceable standards for contaminants which may affect the aesthetic quality of the water.

c. The NPDWR and NSDWR apply to both surface water and ground-water systems.

**4-16. SAMPLING EQUIPMENT**

a. Under normal circumstances, samples are collected directly from a drinking water tap. Therefore, the only equipment that is needed are the sampling containers.

b. The AQAD provides the appropriately prepared containers.

(1) Plastic containers used for several different types of analyses are new and used only once.

(2) VOCs, including trihalomethane (THM), are collected in disposable glass vials. Glass containers for organics analyses should be new. Regardless, the glass bottles should be cleaned as follows:

- (a) Wash with detergent and hot water.
- (b) Rinse with hot tap water.
- (c) Rinse with pesticide grade acetone.
- (d) Air dry.
- (e) Rinse with pesticide grade petroleum ether.
- (f) Air dry.
- (g) Cover with a Teflon-lined cap.

#### 4-17. SAMPLE COLLECTION

The general procedures given below should be followed by both survey personnel and installation personnel who are collecting potable water samples for chemical/radiochemical analyses. Samples for bacteriological analyses have requirements beyond those presented in paragraph 4-16, as discussed in paragraph 4-20.

a. **Sampling location.** The sampling location must be representative of the water system to be monitored. The two types of sampling locations are individual wells and distribution systems supplied by either surface sources or wells.

(1) Individual wells.

(a) Under some circumstances, a sample must be taken directly from a well before the water is treated.

(b) In most cases, there will be a utility spigot near the well pump or the water main that can be used to draw the sample.

(2) Distribution systems.

(a) A representative sampling point in a distribution system is a faucet that is in a good state of repair and is fed by the main water line or a branch water line. Sampling at a dead end should be avoided.

(b) Samples must be collected from several different locations when analyzing for THMs. The sampling locations are as follows:

1. One sample of the raw water prior to any treatment.

2. Three samples at points of moderate to high consumption.

3. One sample at the system's endpoint.

(c) Containers for all other analyses may be filled from the same faucet.

b. **Sample volume.** The volume necessary for each type of analysis is listed in Table 4-1. However, it is always wise to check with individual laboratories concerning the volume required.

c. **Collection procedures.**

(1) Select a clean faucet which does not have an aerator, hose, or other attachment. Avoid swivel faucets if possible.

(2) Allow the water to run at a moderate flow for generally 2 to 3 minutes.

(a) This will clear out water that may have been standing in the building plumbing system and should begin a draw of water from the external water main via the service line.

(b) To assure the withdrawal of water from the main, the temperature may be monitored to determine when flushing is complete.

(c) A drop and then stabilization of the temperature will be noticed, indicating the time to obtain the sample.

(3) Slow the flow rate to a point that the container may be filled without splashing.

(4) Samples for VOCs must be collected carefully to avoid the presence of air bubbles in the containers.

(a) It is best to use a bailer with a bottom check valve or a bladder pump with a low flow rate to collect the samples to assure a minimum of aeration.

(b) Pour sufficient sample into the vial to form a reverse meniscus (rounded surface).

(c) Carefully slide the septum across the top of the vial with the Teflon side toward the water and screw on the cap.

(d) Make sure that no air bubbles remain in the sample by turning the capped vial upside down and tapping the side lightly to force any bubbles to rise.

1. If bubbles are present, repeat the procedure.

2. Do not empty the vial; rather, just add enough water to remove the headspace at the top of the vial.

(e) A few crystals of ascorbic acid must be added to each of the vials to stop the chlorine reaction with organic materials that may be present.

1. The AQAD will normally provide vials with the crystals already in them and a container of 1: 1 hydrochloric acid (HCl).

2. Add 2 drops of the acid solution to each sample vial.

(f) As part of the QC for the analysis for VOCs, at least one travel blank (a vial already filled with water from the USAEHA laboratory) must be returned with each day's sampling group. Keep them with the sample vials at all times in storage, in the field, and in the laboratory. Do not open them.

(5) Containers for all other types of analyses should be filled to the neck.

(a) If a preservative is required, it should be added, and the container capped and shaken to effect mixing.

(b) Containers should be marked with the proper preservative.

(c) In most cases, when acid is added to the sample for preservation, 2 to 3 mL is sufficient to lower the pH to the desired level (less than 2).

(d) Often, small glass ampules of acid are provided by AQAD. Carefully snap off the top and add the entire contents to the sample.

#### 4-I 8. SAMPLE MANAGEMENT

##### a. Containers.

(1) Requests for sample containers should be directed to AQAD at least 30 days before containers are required.

(2) Paragraph 3-2a provides general information on sample containers, and Table 4-I lists the containers appropriate to each type of analysis.

(3) Appendix F details the procedures for submitting sample paperwork.

##### b. Preservation.

(1) Chemicals necessary to preserve the water samples will be provided by AQAD along with sample containers.

(a) Table 4-I lists the proper preservation for each contaminant.

(b) Acid preservatives (sulfuric, nitric, and dichromate in HNO<sub>3</sub>) should be of reagent grade quality so that impurities do not contaminate the sample. The acids will be provided in either bulk quantity or individual ampules depending on the needs of the project officer.

(2) Sometimes the installation will be able to provide the proper preservatives.

(a) Ensure that installation-provided chemicals are of appropriate quality.

(b) This may alleviate some problems with shipping acids by DOT guidelines.

(3) Several drinking water parameters require only cooling for preservation.

c. Labeling. See paragraph 4-5c.

**d. Field sampling form.**

(1) When containers are sent for sample collection by installation personnel, AEHA Form 328-R (Drinking Water Supply Field Data Sheet) must be completed by installation personnel at the time of sample collection. Figure 4-I shows an example of this form. This form will be locally reproduced. A copy for reproduction purposes is located at the back of this TG.

(2) Information such as the date, time, name of sampler, sampling location, and field pH and temperature measurements is required.

**e. Shipping.**

(1) Because several of the drinking water parameters have a maximum holding time of 48 hours, every effort must be made to deliver the samples to the laboratory within 24 hours of collection.

(a) This is usually accomplished through the use of Federal Express overnight delivery.

(b) Air shipment forms and instructions are available from AQAD.

(2) In contrast to wastewater studies, which often use composite sampling, potable water samples for chemical analyses are usually daily individual collections for a given location. Therefore, sampling can most often be scheduled for Monday or Tuesday, allowing samples to arrive at USAEHA laboratories no later than Wednesday.

(a) This will allow the analysis of parameters with 48-hour holding times to be completed before the weekend.

(b) Other parameters may be analyzed at a later time.

(3) If it is necessary for the samples to arrive during the weekend, the laboratories must be notified.

(4) Samples must be analyzed for BOD within 48 hours of collection. The procedure requires incubating diluted samples for 5 days.

(5) If the analyses are begun on Monday or Tuesday, the analyst will be required to work on the weekend in order to measure the final dissolved oxygen (overtime required). Where possible, schedule the collection of BOD samples as to minimize the need for overtime.

**4-I 9. QUALITY ASSURANCE**

a. To ensure the accuracy of the analytical results, QA provisions must be made in both the field and laboratory. Potable water sampling differs from that of most other water sampling in that--

(1) The expected concentrations of parameters are generally much lower.



(2) Any detected contamination potentially presents a more immediate hazard to public health.

b. The nature of this type of sampling demands the implementation of QA measures. A detailed discussion of QA/QC may be found in paragraph 2-4.

#### 4-20. BACTERIOLOGICAL SAMPLING

Although collecting samples for bacteriological analysis is basically the same as for other potable water samples, there are some unique aspects that need to be addressed. In most situations, the sampling and analysis will be performed by the WQED microbiologist or trained Preventive Medicine (PVNTMED) Service personnel from the installation. In some cases, the analysis may be contracted to a commercial laboratory.

##### a. Representative sampling.

(1) If the analyses are to meet compliance requirements, the number of samples collected is based on the population served by the water system. The required number of samples may be determined from the table in 40 CFR 141 (Appendix A, reference 6).

(2) If the samples are for a project, the project officer may determine the number and location of samples to meet the needs of the study. Samples should give a representative view of the system. This means that-

(a) Samples must be collected from several different areas, particularly housing units.

(b) Dead-end lines should also be examined, if they exist, to determine that the proper chlorine residual is being maintained.

##### b. Containers.

(1) Standard containers for bacteriological monitoring of potable water are 125 mL in size and may be either glass or plastic.

(2) The bottles must be sterile and contain 0.1 mL of a 10-percent solution of sodium thiosulfate to quench the chlorine reaction.

(3) Commercially available Whirlpak' bags may also be used.

##### c. Onsite tests.

(1) When bacteriological sampling is performed, the pH and chlorine residual of the wafer must also be tested.

(2) This is usually done with a colorimetric test kit. The WQED warehouse has these kits and instructions for use.

**d. Collection.**

(1) The water samples must be collected in a cautious manner in order not to contaminate the sample.

(a) The faucet does not need to be flamed, but it should be clean and uncorroded.

(b) Remove the container cap, collect the sample, and replace the cap without touching the inside surfaces of either the cap or bottle.

(2) At least 100 mL of water must be collected for each sample.

**e. Shipping precautions.**

(1) Bacteriological samples should be kept cold if analysis will not occur within 1 hour of collection. Samples may be kept in a cooler with ice, or preferably ice packs, until arrival at the laboratory.

(2) If Whirlpak bags are used, care must be taken to secure the bags upright in the cooler. Contamination may occur if the bags are allowed to lay in melted ice water.

(3) Analysis must be performed within 30 hours after the samples have been collected.

**SECTION IV - WATER AND SEDIMENT BIOLOGICAL SAMPLING  
FOR IMPACT DETERMINATION****4-21. INTRODUCTION**

a. When determining the impact on a receiving water from an industrial or domestic wastewater discharge, there are many sampling techniques available. It is generally very helpful in impact studies to determine the physical and chemical water quality as well as the condition of the biotic community.

(1) The purpose of sampling is to determine--

(a) What water quality changes, if any, have occurred.

(b) What effect those changes have on the biological community living in the receiving waters.

(2) The changes in water quality can be subtle and changeable in the receiving water from a discharge. A number of biological characteristics make the biotic communities, which are protected by the discharge permits, excellent indicators of environmental stress.

(3) The biota is an integral part of the ecosystem, and changes in any part of the living or environmental components normally upsets the balance and causes changes throughout the system. These changes are reflected in the community structure. The most sensitive and least mobile organisms will either adapt or perish. The mobile organisms will avoid the stressful situation, adapt, or perish.

(4) The resulting community structure is the sum of the biotic, physical, and chemical interactions.

(a) Impacted communities generally are composed of a small number of species represented by a large number of organisms (low diversity).

(b) Unimpacted communities have many different species with few individuals in a given species (high diversity).

(5) The comparative use of community diversity results in a simple and quantitative summary of pollutional stress.

b. There are many parts of the community that may be sampled in different ways depending on the situation.

(1) The organisms sampled most often are the macroinvertebrates and algae. Representative samples above and below a discharge into a stream (control and impacted samples, respectively) can often indicate the degree of impact on the biotic community of the receiving water.

(2) Discharges to a lake or estuary are much more difficult to quantify, since a control sample is not easily obtained. It is especially difficult to obtain control data on estuaries since the tides are constantly changing the direction the water is moving.

#### 4-22. SURFACE WATER SAMPLING

a. A water sample must represent as closely as possible the source being sampled.

(1) In the case of streams, lakes, reservoirs, estuaries, or the ocean itself, a grab sample could be sufficient if one does not expect much change in water quality over time or distance.

(2) However, if there is a variable discharge entering near the sample location, a time composite is more representative.

b. The samples should be taken, preserved, shipped, and analyzed as discussed in Sections I and II of this chapter.

c. The normal indicators of fecal pollution are FC, fecal strep (FS), and total coliform if shellfish are involved.

(1) If the pollution is relatively recent, the ratio of FC/FS can be used to indicate whether the pollution was from a human source ( $>4$ ) or nonhuman ( $<1$ ).

(2) If the ratio is between 1 and 4, it may indicate a mixture or older human contamination. The die-off rate of FC is faster than FS in water.

d. The samples should be cooled and analyses conducted within 6 hours of collection.

e. The analyses should be performed by a microbiologist, the person qualified to monitor the FC for the sewage treatment plant discharge permit, or the local medical department activity (MEDDAC).

#### 4-23. SEDIMENT SAMPLING

a. Under certain circumstances, sediment samples may be more valuable than water samples in showing the environmental impact on a community. Sampling sediments is useful when pollution--

(1) is suspected at very low levels,

(2) has been detected in the past, or

(3) has been detected on an intermittent basis.

In these cases, water sampling alone may shed very little light on the environmental fate of the pollution.

b. Several pollutants such as pesticides, heavy metals, and several of the organic priority pollutants will accumulate in the sediment. Again, it is important to collect control as well as impacted samples.

c. The sediment may be collected by a variety of methods.

(1) The Ekman dredge and core sampler work well when the sediment is fine and soft, for it is possible to maintain the integrity of the sample.

(2) A Petersen, Ponar, Orange Peel, or Smith-McIntyre sampler is required when the sediment is sandy or hard.

d. Samplers must be properly cleaned, following the methods described in Section I of this chapter, before collecting the sample.

e. If metals are to be analyzed, brass samplers should not be used. Plastic samplers should not be used when analyzing for organics.

f. Sediments should be well mixed before placing in the sample container. A stainless-steel bucket lined with Teflon is the best container in which to mix the sediment.

g. If there is a need to compare the different layers of sediment, the easiest way to sample is to--

(1) Use a core sampler with plastic sleeves cut in half and taped back together.

(2) Fill the tube to the top with sediment, then cap both ends of the tube, marking which end is the top.

(a) If there is water on top of the sediment, it should be drained off. Great care must be taken not to disrupt the sample.

(b) The sediment may later be frozen, the two halves of the tube separated, and the sediment divided however necessary.

## SECTION V - SURFACE WATER

### 4-24. INTRODUCTION

a. Samples must often be collected from ponds, streams, lakes, etc. during a sampling project. Obtaining a representative sample is essential if the resulting data is to be meaningful. Although collection of water samples is easily accomplished, ensuring the water body is well-represented is much more difficult.

b. The project officer must assess the types and amount of changes occurring in the water and adjust his sampling rationale accordingly. This assessment will determine whether a grab sample is sufficient, or a composite (with respect to time, distance, or flow) is required.

### 4-25. SAMPLE COLLECTION

a. Surface liquid samples from shallow depths may be collected by submerging the sample container below the surface of the water. This method is helpful when the sample might be significantly altered during transfer from a sampling device into another container. Examples of analyses which are affected in this manner are grease and oil and VOCs.

b. When a sampling device is used, it should--

(1) Either be disposable or constructed of glass, stainless steel, or Teflon.

(2) Have a capacity of at least 500 mL to minimize the number of times the liquid must be disturbed.

c. In situations where cross contamination is of concern, use of a disposable container or the actual sample container is recommended. The cost of proper cleaning usually exceeds the cost of disposal of otherwise reusable glassware or bottles. This is nearly always true when the cleanup must be accomplished in the field.

d. In some instances, water samples must be collected away from the shore of a water body.

(1) A device which includes a telescoping pole with an adjustable beaker clamp attached to the end is useful for this purpose.

(2) When subsurface samples are required, an at-depth sampler may be necessary. Such an instrument, composed of noncontaminating materials may not be commercially available. It may be necessary to fabricate or modify an existing sampler, such as a Kemmerer-type sampler.

#### 4-26. SAMPLE MANAGEMENT

a. Table 4-1 lists the appropriate sampling containers, preservation requirements, and other information pertaining to water samples.

b. Refer to paragraph 4-5 for a discussion of sample management procedures.

Table 4-1  
Water Sample Collection Requirements

| Analysis  | Minimum (1)<br>Quantity | Container (2)                      | Special<br>Preparation | Preservation                                 | Holding<br>Time (3) |
|---|-------------------------|------------------------------------|------------------------|--|---------------------|
| <b>ORGANICS</b>                                 |                         |                                    |                        |  |                     |
| Acid Extractables                               | 1,000 mL                | G, Teflon-lined Cap                | (11)                   | 4°   | 7 Days (12)         |
| Base/Neutral Extractables                       | 1,000 mL                | G, Teflon-lined Cap                | (11)                   | 4°   | 7 Days (12)         |
| Volatiles or Trihalomethanes (THM)              | 40 mL (x3)              | G, Teflon-lined Cap                | No Head Space, (7)     | 4°, 1:1 HCl to pH < 2                        | 14 Days (13)        |
| Explosives (see parameter listing in Table 7-1) | 1,000 mL                | G, Teflon-lined Cap                | Protect from light     | 4°   | 7 Days              |
| Fuels   | 1,000 mL                | Narrow Neck<br>G, Teflon-lined Cap | None                   | 4°   |                     |
| Pesticides/PCBs                                 | 1,000 mL                | Wide Mouth<br>G, Teflon-lined Cap  | (21)                   | 4°   | 7 Days (12)         |
| Herbicides                                      | 1,000 mL                | G, Teflon-lined Cap                | None                   |  | 28 Days             |
| Total Organic Halides (TOX)                     | 1,000 mL                | G, Teflon-lined Cap                | No Head Space          | 4°, H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 25 Days             |
| Total Petroleum Hydrocarbons                    | 2,000 mL                | G, Teflon-lined Cap                |                        | 4°, HCl-5 mL                                 |                     |

See notes on page 4-37.

Table 4-1  
Water Sample Collection Requirements (Continued)

| Analysis                         | Minimum (1)<br>Quantity | Container (2) | Special<br>Preparation | Preservation                                 | Holding<br>Time (3)    |
|----------------------------------|-------------------------|---------------|------------------------|--|------------------------|
| <b>METALS (16)</b>               |                         |               |                        |  |                        |
| Total Metals (incl Hg, Hardness) | 1,000 mL                | P (20)        | #one (19)              | HNO <sub>3</sub> to pH < 2                   | 6 Months               |
| Dissolved Metals                 | 1,000 mL                | P (20)        | Filtration 0.45 $\mu$  | HNO <sub>3</sub> to pH < 2                   | 6 Months               |
| Chromium VI                      | 250 mL                  | P (20)        | None                   | 4°   | 2 Days (17)            |
| <b>NONMETALS</b>                 |                         |               |                        |  |                        |
| Acidity (4)                      | 250 mL                  | G, P          | None                   | 4°   | 14 Days                |
| Alkalinity (4)                   | 250 mL                  | G, P          | None                   | 4°   | 14 Days                |
|                                  |                         |               |                        |  | 2 Days<br>NPDWR/USAEHA |
| Ammonia (5,4)                    | 250 mL                  | G, P          | None                   | 4°, H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days                |
| BOD (4)                          | 1,000 mL                | G, P          | None                   | 4°   | 2 Days                 |
| BOD, Carbonaceous (4)            | 1,000 mL                | G, P          | None                   | 4°   | 2 Days                 |

See notes on page 4-37

Table 4-1  
Water Sample Collection Requirements (Continued)

| Analysis                           | Minimum (1)<br>Quantity | Container (2)       | Special<br>Preparation | Preservation                                 | Holding<br>Time (3)     |
|------------------------------------|-------------------------|---------------------|------------------------|--|-------------------------|
| <b>NONMETALS (Continued)</b>       |                         |                     |                        |  |                         |
| ROD Soluble (4)                    | 1,000 mL                | G, P                | Filtration (9)         | 4°   | 2 Days                  |
| Bromide (4)                        | 250 mL                  | G, P                | None                   | None   | 28 Days                 |
| Chemical Oxygen Demand (COD) (4)   | 125 mL                  | G, P                | None                   | 4°, H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days                 |
| Chloride (4)                       | 250 mL                  | G, P                | None                   | Room Temperature                             | 28 Days<br>7 Days-NSDWR |
| Chlorine                           | 250 mL                  | G, P                | None                   | None   | Immediately             |
| Color (4)                          | 150 mL                  | G, P                | None                   | 4°   | 2 Days                  |
| Cyanide (4,5)                      | 1,000 mL (6)            | G, P                | None                   | 4°, NaOH to pH > 12 (7)                      | 14 Days                 |
| Cyanide, Amenable (4)              | 1,000 mL (6)            | G, P                | None                   | 4°, NaOH to pH > 12 (7)                      | 14 Days                 |
| Fluoride (4)                       | 125 mL                  | P                   | None                   | None   | 28 Days                 |
| Grease & Oil (4)                   | 1,000 mL                | G, Teflon-lined Cap | None                   | 4°, H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days                 |
| Iodide                             | 250 mL                  | G, P                | None                   | None   | 28 Days                 |
| Iodine                             | 250 mL                  | G, P                | None                   | None   | Immediately             |
| Kjeldahl Nitrogen, Total (TKN) (4) | 250 mL                  | G, P                | None                   | 4°, H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days                 |

See notes on page 4-37.

Table 4-1  
Water Sample Collection Requirements (Continued)

| Analysis                                  | Minimum (1)<br>Quantity | Container (2)       | Special<br>Preparation | Preservation                                 | Holding<br>Time (3) |
|---|-------------------------|---------------------|------------------------|--|---------------------|
| <b>NONMETALS (Continued)</b>              |                         |                     |                        |  |                     |
| Nitrate (NO <sub>3</sub> ) (4,8)          | 125 mL                  | G, P                | None                   | 4"   | 2 Days              |
| Nitrate/Nitrite Nitrogen (4)              | 125 mL                  | G, P                | None                   | 4", H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days             |
| Nitrite (NO <sub>2</sub> ) (4,8)          | 125 mL                  | G, P                | None                   | 4"   | 2 Days              |
| Organic Nitrogen, Total (TON) (4)         | 250 mL                  | G, P                | None                   | 4", H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days             |
| Organic Carbon, Total (TOC) (4)           | 125 mL                  | G, P                | None                   | 4", H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days             |
| pH (4)                                    | 150 mL                  | G, P                | None                   | 4"   | 2 Days              |
| Phenol, Total (4,5)                       | 1,000 mL                | G, Teflon-lined Cap | None                   | 4", H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days             |
| Phosphate, Ortho (4)                      | 150 mL                  | G, P                | Filtration (9)         | 4"   | 2 Days              |
| Phosphate, Total (PO <sub>4</sub> /P) (4) | 150 mL                  | G, P                | None                   | 4", H <sub>2</sub> SO <sub>4</sub> to pH < 2 | 28 Days             |
| Purgeable Organic Halides (POX)           | 1,000 mL                | G, Teflon-lined Cap | No Head Space          | 4"   | 2 Days              |
| Total Dissolved Solids (TDS) (4)          | 250 mL                  | G, P                | None                   | 4"   | 7 Days              |
| Total Suspended Solids (TSS) (4)          | 1,000 mL (10)           | G, P                | None                   | 4"   | 7 Days              |
| Residue, Settleable (4)                   | 1,000 mL                | G, P                | None                   | 4"   | 2 Days              |

See notes on page 4-37.

Table 4-1  
Water Sample Collection Requirements (Continued)

| Analysis                                   | Minimum (1)<br>Quantity | Container (2) | Special<br>Preparation | Preservation                                       | Holding<br>Time (3)     |
|--|-------------------------|---------------|------------------------|--|-------------------------|
| <b>NONMETALS (Continued)</b>               |                         |               |                        |  |                         |
| Total Volatile Solids (TVS) (4)            | 250 mL                  | G, P          | None                   | 4°   | 7 Days                  |
| Total Volatile Dissolved Solids (TVDS) (4) | 250 mL                  | G, P          | None                   | 4°   | 7 Days                  |
| Total Volatile Suspended Solids (TVSS) (4) | 1,000 mL                | G, P          | None                   | 4°   | 7 Days                  |
| Total Solids (TS) (4)                      | 250 mL                  | G, P          | None                   | 4°   | 7 Days                  |
| Silica (4)                                 | 250 mL                  | P             | None                   | 4°   | 28 Days                 |
| Specific Conductivity (4)                  | 250 mL                  | G, P          | None                   | 4°   | 28 Days                 |
| Sulfate (4)                                | 250 mL                  | G, P          | None                   | 4°   | 28 Days<br>7 Days-NPDWR |
| Sulfide (4)                                | 250 mL (x2)             | G, P          | No Head Space          | 4°, 0.5 mL 2N zinc acetate<br>NaOH, pH > 9 1250 mL | 7 Days                  |
| Sulfite (4)                                | 250 mL                  | G, P          | No Head Space          | None   | Immediately             |
| Surfactant (4)                             | 1,000 mL                | G, P          | None                   | 4°   | 2 Days                  |
| TOC, Soluble (4)                           | 125 mL                  | G, P          | Filtration (9)         | 4°, H <sub>2</sub> SO <sub>4</sub> to pH < 2       | 28 Days                 |
| Turbidity (4)                              | 150 mL                  | G, P          | None                   | 4°   | 2 Days                  |

See notes on page 4-37.

Table 4-1  
Water Sample Collection Requirements (Continued)

| Analysis   | Minimum (1)<br>Quantity | Container (2)       | Special<br>Preparation | Preservation               | Holding<br>Time (3) |
|--|-------------------------|---------------------|------------------------|----------------------------|---------------------|
| <b>NONMETALS (Continued)</b>                       |                         |                     |                        |                            |                     |
| Volatile Acids                                     | 1,000 mL                | G, P                | None                   | 4°                         | 28 Days             |
| Dissolved Oxygen (DO)                              | 300                     | G, Teflon-lined Cap | None                   | 4°                         | Immediately         |
| Petroleum Hydrocarbons                             | 1,000 mL                | G                   | None                   | 4°, 5 mL HCl               | 2 Days              |
| Temperature  |                         | G, P                |                        | None                       | Immediately         |
| <b>RADIO CHEMICALS (18, 22)</b>                    |                         |                     |                        |                            |                     |
| Gross Alpha  | 1 gal                   | Plastic Cubitainer  | None                   | HNO <sub>3</sub> to pH < 2 | NA                  |
| Gross Beta   | 1 gal                   | Plastic Cubitainer  | None                   | HNO <sub>3</sub> to pH < 2 | NA                  |
| Tritium  | 1 qt                    | Plastic Cubitainer  | None                   | None                       | NA                  |
| Strontium-90 (14)                                  | 1 gal                   | Mastic Cubitainer   | None                   | HNO <sub>3</sub> to pH < 2 | NA                  |
| Uranium (14)                                       | 1 gal                   | Plastic Cubitainer  | None                   | HNO <sub>3</sub> to pH < 2 | NA                  |
| Radium-226 (14)                                    | 1 gal                   | Plastic Cubitainer  | None                   | HNO <sub>3</sub> to pH < 2 | NA                  |
| Radium-228 (14)                                    | 1 gal                   | Plastic Cubitainer  | None                   | HNO <sub>3</sub> to pH < 2 | NA                  |
| Photon Emitters by Gamma<br>Spectral Analysis (14) | 1 gal                   | Plastic Cubitainer  | None                   | HNO <sub>3</sub> to pH < 2 | (15)                |

NOTES: If certain parameters are combined and volumes are limited, less volume can be used but the laboratory MUST be consulted.

- (1) If the volume of sample is limited, contact laboratory for assistance. Sometimes several parameters can be combined in the same container.
- (2) G - Glass, P = Polyethylene
- (3) Nonmetals Analysis Branch (NMAB) will abide by holding times when mandated by law.
- (4) Permit methods are available for these parameters. Specify on the memorandum if samples are associated with regulation or permit (NPDES, NPDWR, NSDWR).
- (5) Filtration of the sample may result in a loss of the element of interest. In the case of low level ammonia, filtration may contaminate the sample.
- (6) If samples are from industrial processes or are unusually dirty, take two samples from each site for the parameters indicated. Special cleanup procedures and method development may require additional sample.
- (7) If samples contain chlorine, add 0.6 gram (g) ascorbic acid to each sample.
- (8) Do not request these parameters unless absolutely necessary. The analysis of samples for nitrite/nitrate nitrogen ( $\text{NO}_2, \text{NO}_3$ ) should be suitable for most applications.
- (9) Provide filter blanks along with samples. Filters should be washed before use. It is preferable to have the laboratory check the filters for contamination before the scheduled date of the study.
- (10) If the amount of suspended solids is high, less sample can be taken.
- (11) If residual chlorine is present, add 80 milligram (mg) sodium thiosulfate per liter of sample.
- (12) Samples must be extracted within 7 days and analyzed within 40 days.
- (13) Holding time for acetone/acrylonitrile (if requested) is 3 days. Holding time for THM is 28 days.
- (14) Specific analyses are usually performed only when screening limits for the gross beta and gross alpha analyses are exceeded.
- (15) Samples which have short half-lives will require expeditious shipping and analysis. These samples will be handled on a case-by-case basis.
- (16) See Appendix B for additional information concerning metals analysis in water.
- (17) Samples requiring hexavalent chromium ( $\text{Cr}^{+6}$ ) analyses must arrive at the laboratory no later than Wednesday. The advanced memorandum must specify  $\text{Cr}^{+6}$  and state "priority sample."
- (18) One gallon cubitainer is sufficient for analysis of all radioactive parameters except tritium.
- (19) State regulation may require that ground-water samples be filtered before analysis for metals.
- (20) If necessary, glass containers may be used if prewashed and rinsed with  $\text{HNO}_3$  and deionized water. The cap must be Teflon-lined. Reference: EPA-600/4-79-020, rev March 1983, Table 1, P.XVI.
- (21) If residual chlorine is present, add 40 mg sodium thiosulfate per liter.
- (22) Contact Chief, Radiological Analysis Branch (RAB) for sampling information and approval.

## CHAPTER 5 COLLECTION OF SOIL SAMPLES

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### 5-1. INTRODUCTION

a. This chapter provides guidance on the sampling of soils and sediments for the determination of physical properties, hazardous constituents, or characteristics.

b. In some cases, analytical results will characterize soils or sediments as hazardous wastes (HWs), but the methods used to collect environmental samples differ from those used to collect waste samples. (Chapter 6 discusses the sampling of solid and hazardous wastes, which are often containerized and may be of a widely variable nature.)

c. Before going into the field use checklists.

(1) Figures 5-1 and 5-2, shown on pages 5-9 and 5-10, contains a checklist that identifies the specific tests that appear to be of value for the site of interest.

(2) Figures 5-3 and 5-4, shown on pages 5-11 and 5-12, contains a checklist of around 20 items related to QA and QC of the sampling process.

### 5-2. TYPES OF SOIL SAMPLES (Appendix A, references 25, 42, 51, and 54)

The methods used to collect soil samples in the field depend primarily on the purpose for which the sample is needed. Soil samples can be characterized into four main categories:

a. **Type 1.** Samples taken for Unified Soil Classification System (USCS) or U.S. Department of Agriculture (USDA) classification only.

b. **Type 2.** Samples taken for recompacted permeability (noncohesive soils), moisture density relationship, or the permeability at Proctor Density (standard or modified).

c. **Type 3.** Samples taken for in-situ permeability.

d. **Type 4.** Samples taken for chemical analysis.

### 5-3. EQUIPMENT

a. **Surface sampling.**

(1) Soil samples are generally collected with scoops or shovels when sampling on the surface. A variety of other devices may also be used.

(2) Bulb planters are often useful and are easily obtained. These are especially helpful when a somewhat undisturbed sample is desired.

(3) A cork borer may be appropriate if a small amount of sample is required (such as for PCB analysis).

**b. Sediment sampling.**

(1) Sediments may be collected in the same manner used for sludges (see paragraph 6-3); however, a number of additional factors must be considered.

(a) Streams, lakes, and impoundments will probably demonstrate significant variations in sediment composition with respect to distance from inflows, discharges, or other disturbances. It is important, therefore, to document exact sampling locations.

(b) The presence of rocks, debris, and organic material may prevent the use of some devices or require modification to them.

(2) Further information regarding sediment sampling is contained in paragraph 4-23.

**c. Subsurface sampling.** When sampling subsurface soils, Shelby tubes or split spoons are often used in conjunction with drilling equipment. This may be in the form of a power-driven hollow stem auger, or, for more shallow drilling, a hand-held rotary auger. Paragraph 5-4 discusses collection methods specific to the types of soil analyses.

**5-4. COLLECTION METHODS**

This paragraph describes collection methods specific to the types of soil analyses

**a. Type 1.**

(1) When conducting subsurface sampling, soils taken for Type 1 analysis may be collected with a Shelby tube/split spoon or from the auger cuttings.

(a) The Shelby tube/split spoon is used if the soil has multiple strata or if the project officer needs to know the depth of the sample within a few inches.

(b) Auger cuttings are acceptable if the soil is relatively uniform and if less accuracy for the depth of the sample is tolerable.

(2) Approximately ½ kilogram (kg) of soil is required for these analyses.

(3) When sampling from soil pits or from shallow depths, a shovel or trowel may be used.

**b. Type 2.**

(1) Soils taken for Type 2 analysis can also be collected with a Shelby tube/split spoon or from the auger cuttings.

(2) The discussion for the Type 1 sample is pertinent for these analyses; however, a minimum of 4 kg of soil is required. Thus, samples taken with Shelby tubes/split spoons require either--

(a) Continuous sampling in thick horizons, or

(b) Drilling multiple holes or soil test pits at each location to obtain enough soil when thin strata are encountered.

**c. Type 3.**

(1) Soils taken for Type 3 analysis must be collected using a Shelby tube/split spoon. These devices were designed to produce samples which have undergone minimal disturbance.

(2) When possible, the Shelby tube, because it disturbs the sample less, should be chosen over the split spoon.

(3) Type 3 analysis can also be performed with an air entry permeameter, available from WQED.

**d. Type 4.** Samples taken for purposes of chemical analysis require adherence to much more stringent procedures if accurate results are to be achieved.

**(1) Sampling devices and utensils.**

(a) We use split/spoon samplers for sub-surface soil samples.

(b) For smaller-scale projects, it is desirable to take along extra sampling equipment to avoid the need to decontaminate in the field and risk cross contamination. This equipment must be thoroughly cleaned before departing to the field and wrapped (using aluminum foil or other suitable material).

**(2) Avoiding cross contamination.**

(a) An important way to reduce the risk of cross contamination is to begin sampling at the point suspected to be least contaminated and proceed to the most contaminated.

(b) Effective cleanup practices must be followed to prevent contaminants from reaching the sample material by exposure to equipment, containers, or sampling personnel.

(c) If extra equipment is not brought along, sampling materials must be decontaminated in the field. The type of cleaning required will depend upon the type of analyses needed.

(d) Assistance in determining the correct cleaning procedures or solutions can be obtained from the laboratory branch which will perform the analyses. However, the procedures discussed in paragraph 5-5 are generally appropriate.

## 5-5. DECONTAMINATION PROCEDURES

### a. General.

(1) Cross contamination is not an issue for Type 1, 2, or 3 samples, but simple operational cleanliness is recommended. This entails using a wire brush to clean the auger between bore holes and using a clean sampling device between samples.

(2) Prevention of cross contamination in Type 4 samples is essential, since any contamination may significantly alter the analytical results. The decontamination of large equipment, sampling devices, utensils, and sample containers is an essential part of QC in a sampling project.

### b. Equipment.

(1) Equipment decontamination should be performed in an area which is not near boreholes, monitoring wells, or other sampling devices.

(2) The auger, rod, and all tools which come in contact with the soil must be--

(a) Rinsed with pressurized water between each borehole.

(b) Washed with laboratory-grade soap and water

(c) Then rinsed several times with clean water. It is preferable to follow this with a distilled water rinse.

(3) A solvent rinse should not be applied to large equipment.

### c. Sampling devices and utensils.

(1) The sampling device (shovel, split spoon, etc.) must be cleaned between each sample. In addition, all utensils used for collecting, homogenizing, and compositing samples must be thoroughly cleaned between samples.

(2) Requirements for decontamination should be determined when the sampling plan is developed. Consultation with the appropriate laboratory is encouraged.

(3) Decontamination is not required between sampling points when discreet samples from these points are going to be composited.

(4) Devices and utensils must be--

(a) Washed with detergent and water.

(b) Rinsed with distilled/deionized water.

(c) Rinsed with a high-purity solvent (discussed in paragraph d below).

(d) Then rinsed again with distilled/deionized water.

**NOTE:** All decontamination wash solutions must be collected and disposed of properly. One sample may be collected from the wash solution and analyzed to determine HW classification. Typically we also use a sample of the final rinse to indicate effectiveness of decontamination for QC purposes,

**d. Decontamination solvents.** Several factors determine which decontamination solvents are most appropriate in a sampling event.

(1) The targeted parameters will most often dictate the solvent to be used; however, other factors to be considered are the construction material of the sampling device and the relative availability, cost, and hazardous nature of the solvents.

(2) If metals are to be analyzed, an acid rinse is desirable in order to lower the pH and facilitate metals removal. A 1 O-percent HNO<sub>3</sub> solution is generally used for this purpose.

(3) For organics, pesticide grade acetone is an effective solvent, provided it is not a targeted parameter in the study. Acetone is commonly used because of its availability and relatively low cost.

(4) Pesticide grade isopropanol is also an accepted decontaminating solution.

(5) Methanol is considered to be more hazardous to use in the field, and is generally not recommended for field decontamination (Appendix A, reference 45).

(6) Hexane and petroleum ether are not as effective as rinsing agents because they do not mix well with water. These solvents are not recommended unless equipment has been thoroughly dried (Appendix A, reference 45).

**e. Rinse water.** There are several types of treated water which are used in sample bottle preparation and in the cleaning and decontaminating of sampling equipment in the field.

(1) Tap water from a water treatment system is acceptable when cleaning with soap or detergent. Untreated water should not be used in place of tap water.

(2) A distilled/deionized water rinse is an important follow-up step to both the detergent wash and the solvent rinse (performed only after the solvent has completely evaporated). This removes residual detergent and traces of the solvent from the sampling equipment.

(3) Distilled water used in water coolers is not acceptable for field decontamination procedures, except when specified.

(4) Deionized water is that which has been passed through a deionizing resin column, which removes heavy metals and other inorganic compounds. This may be available from laboratories or water treatment plants.

(5) Organic-free water is a higher-grade water which has been treated for the removal of organics as well as for deionization. This is accomplished by passing the water through activated carbon units. This is a desirable rinse water when the analytes are trace organics.

f. **Contain&s.** Contact AQAD for containers.

#### 5-6. SAMPLE MANAGEMENT

General information regarding sample management may be found in Chapter 3. It is important that procedures for labeling, documentation, and chain-of-custody be followed. The following is a discussion of management procedures specific to soil sampling.

a. Soil samples should be homogenized in the field using mixing pans and spatulas or similar utensils. The soil should then be placed into the appropriate containers (see Table 5-7) and labeled immediately.

b. When analyzing for VOCs, samples should not be homogenized, but should be containerized as rapidly as possible, avoiding excessive agitation. Containers should be filled as full as possible and refrigerated immediately.

c. When sampling for explosives and some metal compounds, certain procedures must be followed before samples are submitted to the laboratory for analysis. These include drying and grinding the soil. They are performed in the USAEHA Soils Laboratory. Appendices G and H fully detail these procedures.

d. Samples are usually split in the field at the time of collection. Although this consumes more time in the field, it--

- (1) Is preferable to rehandling the sample after shipment to the laboratory.
- (2) Saves steps in chain-of-custody tracking.
- (3) Better assures that the samples will remain refrigerated until receipt in the laboratory.

Guidance on sample splitting is summarized in Table 5-1 . Specific questions must be referred to the appropriate laboratory.

#### 5-7. QUALITY ASSURANCE/QUALITY CONTROL

a. Any change in standard procedures for field collection of soil samples must be justified, described, and approved by the appropriate project officer.

b. AEHA Form 329-R (Sample Alteration Form) can be used for this purpose. Figure 5-5, on page 5-13, shows an example of this form. This form will be locally reproduced. A copy for reproduction purposes is located at the back of this TG.

c. Soil sampling personnel should be aware of a field audit and ensure that soil sampling and other QA/QC procedures are being followed.

d. Figure 5-6, on page 5-14, contains a field audit checklist and field personnel should review this checklist periodically.

e. Any corrective action required as a result of a field audit should be implemented immediately.

f. Resampling may be required if analytical results fall outside the acceptable limits specified the acceptable limits specified by the AQAD.

g. AEHA Form 331 -R (Field Corrective Action Form) can be used to identify and correct the problems when resampling is required. Figure 5-7, on page 5-1 5, shows an example of this form. This form will be locally reproduced. A copy for reproduction purposes is located at the back of this TG.

h. AEHA Form 332-R (Directorate of Laboratory Sciences Corrective Action Form) may be used to address laboratory problems. Figure 5-8, on page 5-1 6, shows an example of this form. This form will be locally reproduced. A copy for reproduction purposes is located at the back of this TG.

## 5-8. SAMPLE TRANSPORT

Samples must usually be stored and transported so that moisture loss is minimized. When dried beyond 15 bars soil matrix tension, samples change in structure in a way that may affect the final results. Since no visual method has been developed to determine when 15 bars matrix tension is achieved, soil samples must be wrapped in plastic or air-tight containers for shipment.

a. **Type 1.** Samples collected for this type of analysis may be stored and transported in any container that will provide protection against significant loss of moisture. Transportation of these samples is not restricted. However, if these samples are to be transported commercially, the container must meet the minimum requirements of the transporting agency.

b. **Type 2.** The requirements for the storage and transportation of samples collected for this type of analysis are the same as that for the Type 1 analysis.

c. **Type 3.** Samples collected for this analysis must be preserved and sealed in containers that protect the sample from vibrations, shocks, and temperature extremes.

(1) When the project officer personally transports samples by car or truck, the samples need only to fit snugly into cardboard boxes or similar containers that prevent the samples from bumping, rolling, dropping, etc.

(2) Samples, which are to be transported by methods other than outlined above, need to be packed in wooden boxes or crates that provide cushioning and insulation for each sample.

(a) The cushioning material (sawdust, rubber, polystyrene, or material with similar resiliency) should completely encase each sample.

(b) The cushioning between the samples and walls of the shipping containers should have a minimum thickness of 1 inch (25 millimeter (mm)). A minimum thickness of 2 inches (50 mm) should be provided on the container floor.

**d. Type 4.**

(1) If Shelby tubes are used, both ends must be secured for transport using heavy tape or a similar material.

(a) The sample will then have to be extracted from the tube in the USAEHA Soils Laboratory. This is accomplished by--

1. Cutting the tube into sections and loosening the sample by pounding the tube on a hard surface.

2. Then extracting the soil by pushing at one end of the tube, being careful not to contaminate the sample.

(b) Drying and grinding procedures are then performed, if required (see Appendix H).

(2) Assistance in selecting the appropriate handling procedure and sample container can be obtained from Table 5-I or AQAD. Sample size problems should be directed to the appropriate laboratory branch.

**e. Shipping requirements.** Requirements for shipping environmental samples are provided in Appendix D.

|   |
|---|
| <p><b>Soil Physical Properties</b></p> <p><input type="checkbox"/> Color ignition test</p> <p><input type="checkbox"/> Extrastructural crack tests</p> <p><input type="checkbox"/> Fragipan identification</p> <p><input type="checkbox"/> Cementation test</p> <p><input type="checkbox"/> Bulk density</p> <p><input type="checkbox"/> Pocket penetrometer test</p> <p><input type="checkbox"/> Soil temperature regime characterization</p> <p><input type="checkbox"/> Soil moisture</p> <p><input type="checkbox"/> Water table estimation</p> <p><input type="checkbox"/> Available water capacity</p> <p><input type="checkbox"/> Saturated hydraulic conductivity class estimation</p> <p><input type="checkbox"/> Soil drainage class placement</p> <p><b>Soil Engineering Properties</b></p> <p><input type="checkbox"/> Unified (ASTM) texture</p> <p><input type="checkbox"/> Atterberg limits</p> <p><input type="checkbox"/> Shear strength</p> <p><input type="checkbox"/> Shrink-swell tests</p> <p><input type="checkbox"/> Corrosivity characterization</p> |
|---|

Figure 5-I. Checklist of Soil Physical and Chemical Property Sampling and Field Test Procedures

**Soil Chemical Properties**

- Organic matter ignition test
- Cation exchange capacity/exchangeable acidity
- pH
- Redox potential
- Electrical conductivity
- Clay minerals-nitrobenzene test
- Calcium carbonate-HCl test
- Soluble salts-chloride and sulfate
- Gypsum acetone test
- Iron oxides-ignition and streak tests
- Manganese-streak and hydrogen peroxide tests
- Sampling for soil microbiota

Figure 5-2. Checklist of Soil Physical and Chemical Property Sampling and Field Test Procedures  
(Continued)

Check first column to identify needed items. Check second column when item has been obtained and packed prior to leaving for the field.

**Forms**

- List of sample locations where duplicates and other QA samples are to be taken
- Sample alteration form (AEHA Form 329-R), multiple copies

**Field audit checklist (Figure 5-6)**

- Field corrective action form (AEHA Form 331 -RI)

**QA Samples (check types specified in QA Plan)**

*Double-Blind Samples*

- Field evaluation samples (FES)
- Low level field evaluation samples (LLFES)
- External laboratory evaluation samples (ELES)
- Low level external laboratory evaluation samples (LLELES)
- Field matrix spike (FMS)
- Field duplicate (FD)
- Preparation split (PS)

*Single-Blind Samples*

- Field rinsate blanks (FRB)-also called field blanks, decontamination blanks, equipment blanks, and bank blanks
- Preparation rinsate blank (PRB)-also called sample bank blanks
- Trip blank (TB)-also called field blank

Figure 5-3. Soil Sampling Quality Assurance/Quality Control Checklist

|  |
|--|
| <p><b>Sample Preservation and Transport</b></p> <ul style="list-style-type: none"><li>- Chest of 6-pack cooler</li><li>- Ice</li><li>- Max/Min thermometer</li></ul> <p>_____ Chain-of-custody forms and seals</p> <p><u>f</u> <u>o</u> <b>Shipping</b> <u>s</u></p> <p><u>Analytical</u> analysis request forms, if different from chain-of-custody forms.</p> <p><b>Documentation</b></p> <p><u>V</u> <u>e</u> <b>Decontamination</b> <u>I</u></p> <p><u>Wash</u> solution(s)-should be specified in Sampling Plan</p> <p><u>Garden</u> spray cans for wash fluids</p> <p><u>Rinse</u> solutions (acetone, deionized water)</p> <p>_____ Labels for containerized Wastes (solid or liquid)</p> |
|--|

Figure 5-4. Soil Sampling Quality Assurance/Quality Control- Checklist (Continued)

**SAMPLE ALTERATION FORM**  
*The Proponent of this form is the Waste Disposal Engineering Division.*

|  |             |
|--|-------------|
| <small>NOTE: For use of this form see USAEHA TG 155</small>                        |             |
| Project Name and Number: _____   |             |
| Materials to be Sampled: _____   |             |
| Measurement Parameter: _____   |             |
| STANDARD PROCEDURE FOR FIELD COLLECTION AND LABORATORY ANALYSIS (cite reference:), |             |
| 1.   | _____       |
| 2.   | _____       |
| 3.   | _____       |
| 4.   | _____       |
| REASON FOR CHANGE IN FIELD PROCEDURE   |             |
| 1.   | _____       |
| 2.   | _____       |
| 3.   | _____       |
| 4.   | _____       |
| VARIATION FROM FIELD PROCEDURE   |             |
| 1.   | _____       |
| 2.   | _____       |
| 3.   | _____       |
| 4.   | _____       |
| SPECIAL EQUIPMENT, MATERIALS, OR PERSONNEL REQUIRED                                |             |
| 1.   | _____       |
| 2.   | _____       |
| 3.   | _____       |
| 4.   | _____       |
| Initiator's Name _____   | Date: _____ |
| Project Approval: _____  | Date: _____ |
| Laboratory Approval: _____   | Date: _____ |
| Sample Control Center _____  | Date: _____ |

AEHA Form 329-R, 1 FEB 93 (HSHB-ME-S)

Figure 5-5. Example of a Sample Alteration Form

|   |
|---|
| <p><b>Records to Inspect</b></p> <p><input type="checkbox"/> Chain-of-custody forms</p> <p><input type="checkbox"/> Analytical analysis request forms (if different from Chain-of-custody forms)</p> <p><input type="checkbox"/> Sample tags</p> <p><input type="checkbox"/> Site description forms</p> <p><input type="checkbox"/> Log books</p> <p><b>Sampling Procedures to inspect</b></p> <p><input type="checkbox"/> Equipment</p> <p><input type="checkbox"/> Techniques</p> <p><input type="checkbox"/> Decontamination</p> <p><input type="checkbox"/> Collection of duplicate and field blank samples</p> <p><input type="checkbox"/> Security</p> <p><input type="checkbox"/> Sample storage and transportation</p> <p><input type="checkbox"/> Containers</p> <p><input type="checkbox"/> Contaminated waste storage and disposal</p> <p><input type="checkbox"/> Site description form entries</p> |
|---|

Figure 5-6. Field Audit Checklist

**FIELD CORRECTIVE ACTION FORM**  
*The Proponent of this form is the Waste Disposal Engineering Division*

*NOTE: For use of this form see USAEHA TG 165.*

Project Name and Number: \_\_\_\_\_

Sample Data Involved: \_\_\_\_\_

Measurement Parameter(s): \_\_\_\_\_

Acceptable Data Range: \_\_\_\_\_

---

**I. PROBLEM AREAS REQUIRING CORRECTIVE ACTION**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

---

**II. MEASURES REQUIRED TO CORRECT PROBLEMS**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

---

**III. METHODS OF DETECTING PROBLEMS**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

---

Initiator's Name \_\_\_\_\_ Date: \_\_\_\_\_

Project Approval: \_\_\_\_\_ Date: \_\_\_\_\_

Laboratory Approval: \_\_\_\_\_ Date: \_\_\_\_\_

QA Officer/Reviewer: \_\_\_\_\_ Date: \_\_\_\_\_

Sample Control Center: \_\_\_\_\_ Date: \_\_\_\_\_

**EXAMPLE**

AEHA Form 33 I-R, 1 FEB 93 (MSHB-ME-5)

**Figure 5-7. Example of a Field Corrective Action Form**

DIRECTORATE OF LABORATORY SCIENCES CORRECTIVE ACTION FORM

NOTE For use of this form see USAEHA TG 155

1. DATE(S): \_\_\_\_\_ REPORT NO. \_\_\_\_\_

2. FILED BY: \_\_\_\_\_

3. PROBLEM(S): \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

4. WHAT DATA WAS IMPACTED? (N/A - IF NONE) \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

5. MAJOR AREAS/SITUATIONS INVESTIGATED. \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

6. CONCLUSIONS OF INVESTIGATION: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

7 CORRECTIVE ACTIONS TAKEN - DATE \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

BRANCH APPROVAL: \_\_\_\_\_ DATE: \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_

DIVISION APPROVAL: \_\_\_\_\_ DATE: \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_

DIRECTOR APPROVAL: \_\_\_\_\_ DATE: \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_

FOLLOW-UP INSPECTION FINDINGS: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

INSPECTOR: \_\_\_\_\_ DATE: \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_

AEHA Form 332-R, 1 FEB 93 (HSHB-ML-A)

Figure 5-8. Example of a Directorate of Laboratory Sciences Corrective Action Form

Table 5-1  
Soil Sample Collection Requirements

| Analysis                      | Minimum (7)<br>Quantity | Container (8)                | Special<br>Preparation             | Preservation | Holding<br>Time                         |
|-------------------------------|-------------------------|------------------------------|------------------------------------|--------------|---|
| <b>ORGANICS</b>               |                         |                              |                                    |              |   |
| Acid-Ext., Base-Ext.          | 32 oz                   | G                            |                                    | 4 °C         | 17 Day Extraction,<br>40 Day Analysis   |
| TCLP Acid Ext., TCLP Base Ext | 16 oz                   | G                            |                                    | 4 °C         | 14 Day Extraction.<br>40 Day Analysis   |
| Volatiles                     | 1 oz                    | G                            | Minimum Head Space                 | 4 °C         | ASAP Extraction,<br>14 Day Analysis     |
| TCLP Volatiles                | 4 oz                    | G                            |                                    | 4 °C         |   |
| Explosives                    | 100 g                   | Wide Mouth G<br>w/Teflon lid | Protect from<br>Sunlight, see (12) |              | > 30 Days                               |
| Pesticides (13,151            | 32 oz                   | Wide Mouth G<br>w/Teflon Lid |                                    | 4 °C         | 7 Day Extraction,<br>< 30 Day Analysis  |
| TCLP Pesticides (13)          | 32 oz                   | Wide Mouth G<br>w/Teflon Lid |                                    | 4 °C         | 14 Day Extraction.<br>< 30 Day Analysis |
| PCBs                          | 40 g                    | G w/Teflon Lid               |                                    | 4 °C         |   |
| Herbicides (13)               | 32 oz                   | G w/Teflon Lid               |                                    | 4 °C         | < 7 Day Extraction<br>< 30 Day Analysis |
| TCLP Herbicides (13)          | 32 oz                   | G w/Teflon Lid               |                                    | 4 °C         | 14 Day Extraction<br>< 30 Day Analysis  |
| Total Organic Halide (TOX)    | 60 g soil               | P or G, Wide Mouth           | Soils & Sludges: (6.12)            | 4 °C         | 60 Days                                 |

See notes on page 5.21.

Table 5-1  
Soil Sample Collection Requirements (Continued)

| Analysis                    | Minimum (7)<br>Quantity | Container (8)                    | Special<br>Preparation       | Preservation | Holding<br>Time |
|-----------------------------|-------------------------|----------------------------------|------------------------------|--------------|-----------------|
| <b>METALS (1)</b>           |                         |                                  |                              |              |                 |
| Total Metals                | 32 oz.                  | Wide Mouth G (17)                | Soils & Sludges: (6,12)      | Cool (14)    | 6 Months        |
| TCLP Toxicity               | 500 g (2)               | Wide Mouth G (17)                | SAMPLES MUST NOT<br>BE DRIED |              | Cool (14)       |
| <b>RADIOACTIVITY (5, 4)</b> |                         |                                  |                              |              |                 |
| Gross Alpha                 | 500 g                   | 10" x 10"<br>Doubled Ziplock Bag |                              | None         | NA              |
| Gross Beta                  | 500 g                   | 10" x 10"<br>Doubled Ziplock Bag |                              | None         | NA              |
| Iranium                     | 500 g                   | 10" x 10"<br>Doubled Ziplock Bag |                              | None         | NA              |
| Photon Emitters             | 500 g                   | 10" x 10"<br>Doubled Ziplock Bag |                              | (3)          | (3)             |
| Tritium                     | 500 g                   | Glass w/Teflon Lid               | SAMPLE MUST NOT<br>BE DRIED  | None         | NA              |

See notes on page 5-21.

Table 5-I  
Soil Sample Collection Requirements (Continued)

| Analysis                              | Minimum (7)<br>Quantity | Container (8)      | Special<br>Preparation  | Preservation | Holding<br>Time |
|---------------------------------------|-------------------------|--------------------|-------------------------|--------------|-----------------|
| NONMETALS (2, 9, 10)                  |                         |                    |                         |              |                 |
| Acidity (ACID)                        | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Alkalinity (T-ALK)                    | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Ammonia-nitrogen (NH <sub>3</sub> /N) | 60 g soil               | P or G, Wide Mouth | (16)                    | 4 °C         | 60 Days         |
| Base Saturation % (%BS) (11)          | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Carbon, Total (TC)                    | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Carbon, Total Organic (TOC) (11)      | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Cation Exchange Capacity (CEC) (11)   | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Chemical Oxygen Demand (COD)          | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: 16,121 | 4 °C         | 60 Days         |
| Chloride (Cl) (11)                    | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Conductivity (COND) (11,141)          | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: 16,121 | 4 °C         | 60 Days         |
| Exchangeable Acidity (EXACID) (11)    | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Exchangeable Cations (EXCAT)          | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Fluoride (F)                          | 60 g soil               | P or G, Wide Mouth | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |

See notes on page 5-21.

Table 5-1  
Soil Sample Collection Requirements (Continued)

| Analysis  | Minimum (7)<br>Quantity | Container (8)                              | Special<br>Preparation  | Preservation | Holding<br>Time |
|---|-------------------------|--|-------------------------|--------------|-----------------|
| <b>NONMETALS (2, 9, 10) (Continued)</b>                           |                         |  |                         |              |                 |
| Grease & Oil (G & O)  | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Kjeldahl Nitrogen, Total (TKN) (11)                               | 60 g soil               | P or G, Wide Mouth                         | (16)                    | 4 °C         | 60 Days         |
| Moisture (MOIST)  | 60 g soil               | P or G, Wide Mouth<br>Airtight. Preweighed | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Nitrite/Nitrate Nitrogen (NO <sub>2</sub> ,NO <sub>3</sub> ) (11) | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Organic Matter, Total (TOM)                                       | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| pH (11)   | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Phenol  | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Phosphate, Organic (PO <sub>4</sub> /ORG) (11)                    | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Phosphate, Total (PO <sub>4</sub> /P)                             | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Phosphorus, Available<br>(PO <sub>4</sub> /A) (11)                | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Sulfate (SO <sub>4</sub> ) (11)                                   | 60 g soil               | P or G, Wide Mouth                         | Soils & Sludges: (6,12) | 4 °C         | 60 Days         |
| Volatile Acids (VOLAC)  | 60 g soil               | P or G, Wide Mouth                         | (16)                    | 4 °C         | 60 Days         |

NOTES: The Soil Samples can be combined into the same container if they are of the same sieve size.

- (1) Refer to Appendix C for more information concerning metals analyses on soils and sludges.
- (2) Samples which are extremely light will require special consideration. Please consult laboratory chemist before sampling.
- (3) Samples which have short half-lives will require expeditious shipping and analysis. These samples will be handled on a case-by-case basis.
- (4) Detection limit varies with half-life, gamma energy, and abundance. For Cesium-137 the detection limit is 0.3 pCi/gm.
- (5) Additional analyses can be performed on request. Contact appropriate laboratory for guidance.
- (6) Sludges should be homogenized before distribution to the laboratories.
- (7) Minimum quantities vary according to the amount of water present in the sample. If sample is a soil or dry sludge, provide the laboratory with 60 g of sample per parameter. If sample contains visible amounts of water up to 10 percent of the volume, provide 1 qt of sample per 5 parameters. If the sample visibly contains more than 10 percent water, provide a volume equal to that provided for a water sample.
- (8) P = Polyethylene, G = Glass
- (9) EPA has provided no formal guidance as to holding times; NMAB will abide by EPA guidance when available and mandated by law.
- (10) All sample results will be reported on a dry weight basis unless otherwise requested.
- (11) More than one method available for this parameter. Consult laboratory chemist. Detection limits vary with methods used, Specify detection limit desired.
- (12) Most soils must be air dried and sieved prior to submittal to the laboratory except where volatile components are being analyzed. See Appendix G for Explosives Analysis and/or Metals Analysis and Appendix H for Nonmetals Analysis.
- (13) These parameters are ordinarily included in base-neutral extractable organics analyses. Requirements listed apply to analyses for these parameters exclusively.
- (14) It is not required to preserve these samples by cooling to 4 °C. However, it is generally recommended that samples be kept cool in order to prevent reactions from occurring during handling and transport.
- (15) For certain circumstances (e.g., spill sites), less sample is required. Consult a Pesticides Analysis Branch (PAB) laboratory chemist.
- (16) Homogenization of samples will result in a loss or gain of this parameter. Samples should be disturbed as little as possible, Volatile Acids analyses are performed on sludges only; these should also be disturbed minimally.
- (17) New polyethylene containers may be used for soils and sludge samples. (Reference: EPA SW-846, Chapter Nine, p Nine-47, Field Manual, Volume II, revised 3d Edition, November 1990.)
- (18) Coordination with laboratory is necessary to determine if separate containers are required if analysis of all TCI P parameters is planned.

**CHAPTER 6**  
**COLLECTION OF WASTE SAMPLES**

---

**6-1. INTRODUCTION**

a. This chapter discusses methods for the collection of samples which are "nonenvironmental" in nature, i.e., not collected from the natural environment (water, soil, or sediments).

**NOTE: The sampling of wastewater is not addressed in this chapter. It is included in Chapter 4, since it is more closely related to other types of water sampling.**

b. Sampling of wastes is, however, in keeping with the purpose of this TG. In many cases, sampling of wastes is performed in order to evaluate environmental hazards or to determine the appropriate management practices or disposal methods.

c. Sampling of wastes potentially involves greater hazards than does environmental sampling. Contaminant concentrations may be substantially higher, or hazardous characteristics may be more extreme.

(1) Therefore, safety considerations should not be overlooked when performing this type of sampling.

(2) In addition, Occupational Safety and Health Administration (OSHA) regulations (29 CFR 1910) now require yearly training of all personnel working in HW operations.

(a) USAEHA complies with this requirement

(b) All personnel involved in field activities must complete the 40-hour initial OSHA course followed by an annual 8-hour refresher course.

(3) However, it is not within the scope of this TG to present a detailed discussion of site safety, even though specific cautions are mentioned where relating to certain types of sampling.

(4) Refer to Appendix A, reference 31 for more information on appropriate safety measures and accident prevention.

d. The term "wastes" is used in this chapter rather than "hazardous wastes," because--

(1) The purpose of sampling wastes, more often than not, is to determine whether or not the waste is in fact a HW.

(2) Other wastes may be sampled for determination of marketable value or suitability; for reuse, or for a variety of other reasons.

(3) The sampling procedures in this chapter may also be applied to the sampling of materials which have not yet been determined to be wastes.

e. In general, when collecting and analyzing wastes for purposes of HW characterization, it is preferable to collect a sample at the generation point rather than after the waste has been containerized.

(1) Although this is not always possible, it does result in a more representative sample because the effects of exposure to air, drying and compaction may significantly alter the chemical and physical properties of the waste.

(2) In addition, sampling at the source is often easier than sampling from containers.

f. The following paragraphs discuss the sampling procedures for the collection of wastes and materials, which include solids, semisolids, and liquids. Specific procedures pertaining to drum sampling are also included.

#### 6-2. **SOLIDS** (Appendix A, reference 20)

**a. Characteristics.** The sampling of solid or semisolid wastes is complicated by the structural properties of the substance. For example, the presence of entrapped gases and fluids is often an integral part of the substance and is of consequence in the analytical techniques used on the sample. It is necessary to collect a sample which does not alter this balance.

(1) A solid does not necessarily have uniform characteristics (i.e., contamination, matrix consistency) with respect to distance or depth.

(2) Those portions which form boundaries with the container, define the edges of a pile, or contact the atmosphere do not necessarily represent the material as a whole.

(3) Care must be exercised to prevent aeration or significant changes in moisture content.

(4) Physical strength and density of the material demand sampling devices of significant rigidity and strength.

**b. Sampling devices.** Most commercially available solids sampling devices are steel, brass, or plastic.

(1) In general, use of stainless steel is the most practical, **and** several manufacturers will fabricate their equipment with all stainless-steel parts on a special-order basis.

(2) Another alternative is to have the contact surfaces Teflon-coated.

(3) Some devices, especially those designed for soil sampling, have been chrome- or nickel-plated. These should be avoided in waste sampling, since scratches and flaking of the plating can drastically affect the results of trace heavy metal analyses.

c. Bulk materials. Bulk materials are homogeneous collections of a single identifiable product.

(1) They are usually contained in bags, drums, bins, or hoppers, although on occasion, large amounts of the material may be piled directly on the ground.

(2) Those surfaces exposed to the atmosphere may undergo some chemical alteration or degradation and should be avoided during sample collection.

(3) Since the process producing the bulk material may demonstrate some variation with respect to time, it is advisable to collect a series of samples as one composite to represent the material.

(a) Bulk materials in an unconsolidated state may be collected by a stainless-steel scoop.

(b) When the amount of the material is large, a composite can be collected by the use of a grain thief.

(4) However, it may be difficult and potentially dangerous to use a drum or grain thief sampler when the material is a fine, tightly packed solid. In these circumstances--

(a) The barrel should be dumped onto a plastic or metal sheet and a composite sample should be taken from the top, middle, and bottom.

(b) Care should be taken to contain the waste during sampling and recontainerize the waste after sampling.

### 6-3. SEMISOLIDS

a. Sludges are semidry materials ranging from dewatered solids to high viscosity liquids.

b. Sludges may often be sampled using a stainless-steel scoop when exposed by evaporation, stream rerouting, or other means of water loss.

c. When a liquid layer is sufficiently shallow, the sludge **may be** collected by a device such as a pond sampler or Shelby tube.

d. Sludges which develop in drums can usually be collected by employing the glass rods used for the liquid portion (see paragraph **6-4d(2)(c)3**).

e. If the sludge layer is less than 30 centimeters (cm), corer penetration may damage the container liner or bottom. In this instance, a Ponar grab sampler should be used, provided the opening is large enough to accommodate it.

f. With a small amount of solids below a large amount of liquid, it may be best to mix the contents and treat it as a liquid for sampling purposes

## 6-4. LIQUIDS

### a. General.

(1) Liquids, by their nature, are a relatively easy substance to collect. Obtaining representative samples, however, is more difficult.

(2) Density, solubility, temperature, currents, and other mechanisms cause changes in the composition of a liquid with respect to time and distance. Accurate sampling must be responsive to these dynamics and reflect their actions.

(3) Liquids addressed in this section include surface impoundments (ponds, lagoons) and containerized liquids.

**NOTE: Surface impoundments are not considered surface waters for the purpose of this TG. The collection of samples from surface waters is discussed in Chapter 4, Section V.**

### b. Surface impoundments.

(1) Surface impoundments are complicated by such factors as the location, construction depth, process activity, method of waste entry, rainfall, surface disturbances, and temperatures.

(2) Within the pond, there may be unique sludge depositional patterns, liquid-solid phase separations, liquid-liquid phase separations, and a variety of suspended solids.

(3) An attempt must be made to represent all phases and components in the sample collection. Sampling solids and liquids along the sides and spillways of surface impoundment may **also** be appropriate.

(4) At a minimum, surface impoundments must be sampled in both liquid and solid phases from the shoreline.

(a) For both phases, samples should be collected at several locations around the surface impoundment.

(b) In some special cases (e.g., when the surface impoundment was created by enlarging a natural stream), process waste may be covered with sediment from a storm event. These *cases* need special consideration for sampling.

(5) Results from initial sampling may indicate a need to sample solids and/or liquids away from the shore. This type of sampling is potentially dangerous. The samples may be collected using a sampling platform or rowboat. Samples should be collected at several locations.

(a) For liquid phases, a Kemmerer bottle lowered to a certain depth is appropriate.

(b) For sludges, a clamshell or dredge sampler usually is satisfactory.

(c) Some solids, however, are difficult to sample because their composition and density cause much of the sample to be lost before the sampler is brought to the surface. These cases may require fabrication of scoops fastened to poles which can reach to the desired depth.

**c. Containerized liquids.**

(1) The sampling of tanks, containers, and drums of liquids presents unique problems not associated with those of open-air water bodies (See Appendix I).

(a) The physical size, shape, construction material, and location of access will limit the types of equipment and methods of collection.

(b) Openings on larger vessels may consist of access ports, **manways**, or hatches, while smaller ones may contain taps or bungs.

(2) When liquids are contained in sealed vessels, **gas** vapor pressures build up, sludges settle out, and density layerings develop. The potential for explosive reactions or the release of noxious gases when containers are opened requires considerable safeguards.

(a) The vessels must be opened with extreme caution, using nonsparking tools.

(b) A preliminary check with an explosimeter or an organic vapor analyzer will help in determining the appropriate level of personal protection and may aid in selecting a sampling method.

(3) 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 liquid left undisturbed over time, a sample must be taken that represents the entire depth of the vessel.

(a) Agitation to disrupt the **layers** and rehomogenize the sample is physically difficult and almost always undesirable.

(b) In vessels greater than 1 meter in depth, the method of choice is to slowly lower a suction line from a peristaltic pump in known increments of length. Discrete samples can be collected from various depths and then combined or analyzed separately.

(c) If the depth of the vessel is greater than the lift capacity of the pump, an at-depth water sampler, such as the Kemmerer type, **may be** required.

(d) In situations where the reactive nature of the contents is known, a small submersible pump may be used.

(4) When sampling a previously sealed vessel, a check must be made for the presence of bottom Sludge. This is easily accomplished by measuring the depth to the apparent bottom, then comparing it to the known interior depth. This sludge may be sampled as described in paragraph 6-3.

(5) The sampling of liquids in drums involves many specific procedures and precautions. The following paragraphs fully address this type of sampling.

**d. Drum sampling.****(1) Hazards.**

(a) Drum sampling has the potential to be a very high risk operation. For this reason--

1. These guidelines concerning drum sampling must be adhered to closely.
2. Drums and tanks should be sampled only when necessary to meet regulatory or remediation requirements.

(b) It cannot be overemphasized that all drums must be treated as explosion hazards until proven otherwise.

1. Many materials found in drums may reach the boiling point if exposed to summer weather or full sun. When these drums are opened, gases, vapors, and liquids may be released with violent force.

2. If a drum seems to be empty or almost empty, great care should be exercised **not to** disturb it since potentially hazardous explosive peroxides may have formed during evaporation of the contents.

(c) Project officers should use both common sense and the suspected identity of the drum contents to evaluate the hazard involved.

(d) To minimize the exposure of personnel--

1. Disposable personal protective and sampling equipment should be used.
2. Nonsparking tools should be used to open drums.
3. Rods used for drum sampling may be broken and left inside the drum to prevent injury or contamination.
4. All drums must be resealed after-sampling, either by resealing the bung or installing a plastic snap-on cap.

(e) Drums which are open or have holes in them should **be sampled** only after ambient air monitoring for toxic and explosive vapors has been made at the opening. The characterization of the air in and around the drum should **be made** while using a self-contained **breathing** apparatus (SCBA).

(f) Drums which are open but inaccessible for sampling should be carefully monitored for the presence of explosive and toxic vapors. The work crew should not crawl or walk over drums to reach other drums for sampling.

(g) Sealed drums may be opened for sampling only if the bung can be easily turned using a spark-proof bung wrench.

1. If loosening the bung produces a hissing sound or escaping gases, the operation must be ceased immediately and the crew must leave the area.

2. After the hissing ceases, the operation can be resumed with all appropriate monitoring.

**NOTE: NEVER use a cutting torch to open drums.**

(h) Drums should not be sampled if they are--

1. Sealed and the bung is not easily turned.

2. Bulging, deformed, or corroded.

3. Located in a physically inaccessible place, such as a ravine, gully, or marsh.

## (2) Drum sampling methods.

(a) The most widely used method for sampling drums is with a glass rod, 6 mm to 16 mm in diameter.

1. To use this method--

a. Lower the glass rod into the drum.

b. When the rod has reached the bottom or the desired depth, seal the top of the rod with a stopper or the thumb (be sure to wear gloves during this operation).

c. Withdraw the rod and place it over the sample container.

d. Remove the stopper and drain the contents into the container.

e. After collection of sufficient sample, the rod may be broken up and left in the drum.

2. This method is quick, relatively inexpensive, and requires no decontamination procedures of the sampling equipment. It does, however, have serious drawbacks.

a. Most low density fluids do not hold well in glass rods.

b. A great deal of the potential sample flows out of the bottom of the rod as it is raised from the drum, thereby reducing the chance of obtaining a true representative sample.

3. The following are variations to this technique.

a. Incorporation of a small suction device to the top of the rod.

b. Use of various rod sizes may **also** be helpful (thinner rods will hold the less viscous liquids better).

c. Some success has been reported with rods that have been heated at one end and then drawn to form a much smaller orifice. This enables one to use larger diameter tubing, allowing a greater volume of sample to be collected during each attempt while reducing the material loss from the rod bottom.

d. Another sampling device which has proved successful is the "rod within a rod." The outer glass rod contains the liquid sample and the inner rod lowers to form a plug at the base. These are available in the USAEHA Soils Laboratory.

(b) The Composite Liquid Waste Sampler (COLIWASA) will collect a sample from the full depth of a drum and maintain it in the transfer rod until delivery to the sample bottle.

1. The COLIWASA is difficult to decontaminate properly in the field.

a. Its applicability is, therefore, limited to those cases when a sample of the full depth of the drum is necessary.

b. The COLIWASA can be somewhat modified for this task by making the lift rod of stainless steel or glass, the bottom stopper of Teflon, and the body of glass tubing. In this configuration, the glass rod can be broken into the drum, leaving only the center rod and stopper as potential sources of contamination.

2. In some cases, disposal of the rod by breaking it into the drum may interfere with eventual plans for the removal of the contents. The use of this technique should be cleared with the installation personnel.

3. In many instances, a drum containing waste will have a sludge layer on the bottom.

a. Slow insertion of the sample rod into this layer followed by gradual withdrawal will allow the sludge to act as a bottom plug.

b. The plug can be gently removed and placed into the sample container with a stainless-steel laboratory spoon. These spoons are relatively inexpensive and can be disposed of in the original waste container with the **glass** transfer rod.

c. Acceptable methods for sampling sludges in open-headed drums are to use an Ekman dredge, Ponar sampler, or aluminum hollow rod.

(c) Liquids should never be poured out of a drum into a sample container.

1. This results in a sample which does not accurately represent both the heavier and lighter density components in the drum.

2. This practice is also potentially dangerous, since spills can occur very easily.

3. The use of the glass rods will result in representative samples when there is liquid layering present in the drum. The rods should be used just as if there was one liquid in the drum.

(d) Drums will be sealed by replacing the **bung** cover or using a plastic snap-on cap. If drums are deheaded, they also must be sealed after sampling. **Ring** closures may be used for this purpose, or drums may be overpacked, especially if they are to be transported.

(e) When sampling any quantity of drums, especially when the contents are unknown, it is best to spray-paint a number on each drum. This serves to identify the individual drums and to keep an inventory.

1. Drum numbers should correspond with notes kept in a logbook about each sample taken.

2. The same numbers should also be used to identify sample containers so that analytical results can be matched with drums containing each material.

## 6-5. SPECIAL PROJECTS

### a. General.

(1) The USAEHA performs Some environmental sampling projects which are unique to Army operations. Often these studies involve the collection of samples for reactivity analysis or samples which are potentially reactive in nature.

(a) Such samples may come from a variety of sources at Army ammunition plants, forts, and depots.

(b) Examples of sample Sources at the installation are open burning/open detonation (OB/OD) grounds, explosive waste incinerators, contaminated waste processors, HW landfills, impact areas, and surface impoundments.

(2) Many of the sampling methods involved are covered in other parts of this TG. This section discusses those sources not previously mentioned.

**b. OB/OD grounds.**

(1) In selecting a site **for** residue or soil sampling, contact the **OB/OD** ground operator to determine the sites most recently and/or frequently used.

(a) The site may **consist** Of one large burn pad, a cluster of small burn pads, a burn trench, a flashing pit, a burn cage area, or a demolition trench.

(b) Open burning **pans now** being constructed at selected installations are also considered sampling sites.

(2) All material collected for each sample must be homogenized prior to analysis. Therefore, consider all material randomly collected from each sampling point as one sample.

(3) Obvious piles of ash should be sampled first. Any lumps of fused plastic, glass, metal, paper, cardboard, wood, or other extraneous objects must be removed from the sample. Fine ash or stained soil scattered as a surface veneer should be sampled down to the interface of the material, no deeper than 0.5 inch.

(4) Due to the danger of unexploded ordnance (UXO) being present in the detonation crater, samples should not be taken from inside the crater. Displaced soil can be sampled from around the crater radially from the center at **90-degree** angles.

**c. Waste pits, sumps, and tanks.**

(1) These waste-receiving devices are usually found at process discharge points along an explosive manufacturing or demilitarization facility (e.g., trinitrotoluene (TNT) washout plant, or kill tanks for primer mixes). Other sources may be separator devices, half-tanks, or any number of collection points.

(2) For solids, the use of a clamshell sampler or box-like scoops on poles is recommended.

(3) For liquid samples, a Kemmerer bottle lowered to the correct depth may be applicable, or a dedicated pipette and glass rod may be used.

**d. Incinerators.** Sampling points for incinerators are hearths or Qrates, cyclone precipitators, **electrostatic** precipitators, or bag houses.

(1) Hearth or grate (or pit) ash can be collected from the incinerator tray using a plastic scoop.

(2) The other areas usually have the ash dropping into a barrel or bin. Sampling these ashes should be accomplished as described in paragraph **6-2c**.

e. **Filter media.** Filter media for explosive manufacture is generally one of two types, which are--

(1) **Diatomaceous earth.** Gather the solid sample from the bottom of the backwash sump. (Liquid portion goes to activated carbon filters.)

(2) **Activated carbon.** When the carbon is removed from the filter columns and taken to the OB grounds, sample the pile of carbon as for a **homogeneous** pile (paragraph 6-2c).

6-6. **SAMPLING PROCESS**

a. **General.**

(1) The following procedures are basic instructions that are to be followed with the sampling equipment (containers and pipettes) obtained through AQAD.

(a) If possible, equipment should be obtained directly from USAEHA.

(b) In emergency situations, the installation/facility may use their own equipment (glass pipettes, tubing, and containers).

(2) The AQAD should be contacted at defense switched network (DSN) 584-3269 or commercial 41 O-671 -3269 for information on acceptable equipment.

b. **Equipment.**

(1) The sampling kit provided through USAEHA contains precleaned, ready-to-sample containers, sampling pipettes, and Teflon-lined flexible tubing. If other equipment is being used, its size, type (material), and cleanliness must be approved by AQAD.

(2) The installation/facility must supply all personnel conducting sampling with proper personal protective equipment (PPE). Disposable polyethylene gloves, boots, and Tyvek® suits are recommended. Contact the installation safety office for appropriate PPE.

. . . **IMPORTANT** . . .

**AVOID SPILLS!** Even small amounts of liquid may require specific cleanup procedures that result in the generation of regulated materials/wastes.

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c. **Procedure.**

(1) Begin by putting on the appropriate PPE.

(2) Label sampling container(s) with the following information:

- (a) Date.
- (b) Installation/facility name.
- (c) Type/description/specific location of equipment (to include transformer serial number).

(3) Evaluate the equipment to be sampled.

(a) If the provided equipment does not seem adequate for the necessary sampling, consult USAEHA.

(b) Otherwise, proceed with the appropriate sampling method as described below.

**d. Method.** The type of equipment being sampled will determine the sampling approach to be taken.

**(1) Equipment currently in-use (transformers, capacitors, etc.).** This type of equipment must first be de-energized before sampling. The liquid sample may be taken right from the surface with the pipette or through the vacuum/pressure release valve or other accessible opening with the flexible tubing.

**(a) Pipette method.**

1. Using gloves, submerge approximately two-thirds of pipette into liquid while decompressing rubber ball.

2. Release pressure on rubber ball and allow pipette to fill.

3. Remove and immediately place 5 mL of liquid into sampling container. Return excess liquid to equipment being sampled.

**(b) Tube method.**

1. Insert tubing through opening; ensure that tube reaches the container bottom.

2. Place thumb over end of tubing and carefully withdraw.

3. Using other hand, guide end of tube into sample container

4. **Slowly** release liquid into sampling container filling about three-quarters. Return excess liquid to equipment being sampled.

**(2) Stored drums or equipment.** These contain liquids that may have "settled-out;" therefore, samples must be drawn from the lower depths of the liquid. This is best achieved through using a COLIWASA or drum thief. Follow the "tube method" described above.

**e. Preservation.**

- (1) All samples should be stored in a cool place (**4 °C**) and protected from sunlight.
- (2) Samples should not be stored for more than 24 hours before shipping for analysis.

**f. Packaging/Shipping.** Proper packaging is essential for this process to work.

- (1) All samples should be placed in a Styrofoam packer. (Use the packer that the containers were originally sent in.)
- (2) **Along** with a cold pack/freezer pack (such as Blue Ice -- NOT dry ice, as it cannot be shipped by air), wrap the Styrofoam packer with sufficient paper, etc. to protect during shipment. Place this package in a box or cooler and **Federal Express** overnight mail to USAEHA laboratories. (A box is sufficient for small quantities of samples; a cooler should be used when large numbers of **samples** are being sent.)

**g. Waste disposal.** Waste generated during sampling will include PPE, pipettes and/or tubing.

- (1) The PPE should be disposed of as general waste.
- (2) Pipettes and tubing must be containerized and labeled as possible PCB items, pending analysis, with the sampling date on label. These items may be stored for 30 days until proper identification and disposal procedures are followed.

h. Verification. Within 48 hours, check with the laboratory at USAEHA to assure receipt of samples.

**6-7. WIPE SAMPLING**

a. Wipe sampling is still a developing procedure and should only be used when other procedures are inappropriate. Typical uses of wipe sampling are the identification of surface contamination and the verification of spill cleanups. All methods described below should be coordinated with the laboratories to determine current solvents and procedures.

b. Wipe sampling is most successful on nonporous surfaces; however, porous surfaces such as concrete have been wipe sampled in the past. The recovery rate from these surfaces has not been established. Additionally, some surfaces may leach metals or semivolatile compounds from the actual surface material, not any potential contamination. For these reasons, wipe sampling is recommended only as a screening tool, to establish the presence/absence of contaminants (such as PCBs) and/or in conjunction with background data. Wipe sampling is required for **PCB** spill cleanup verification.

c. Background samples must be taken **from** surfaces similar to the surface of interest. Surface type and any coatings such as paint or lacquer should match as closely as possible. Background samples should be tested for all **parameters** of interest in the study. The number of background samples should be the maximum number feasible to provide statistical significance to data evaluation (up to 100 percent of the actual sample number). An absolute minimum of three background samples is required.

d. Wipe sampling requires the use of a collection material (such as cotton balls) wetted with a solvent and wiped across a standard area of the surface of interest.

(1) The laboratory performing the analysis should be consulted when choosing the collection material. Cotton balls, fiber filters, and swabs can be used, but each has different extraction characteristics which may affect the results. Prewashing and drying the collection material reduce matrix interference and increases accuracy. The sampler should also consider such sampling logistics as how the collection material will be wiped across the surface and placed into the sample container (with metal tongs, plastic forceps, etc.).

(2) The collection material must be wetted (but not soaked) with the appropriate solvent for the parameter of interest. The amount of solvent should be consistent between wipe sample (e.g., 3 milliliters) to control sampling variability. Table 6-1 lists parameters and appropriate solvents: these solvents should be verified with and obtained from the laboratory performing the analyses.

(3) A standard area of 100 cm<sup>2</sup> is recommended for one wipe sample. Disposable templates may be made from manilla folders. Care should be taken not to wipe (and extract) from the template. Wipe the defined area with the wetted collection material both vertically and horizontally.

e. The samples must be placed immediately in a clean glass vial with a Teflon lid and stored at 4°C. A separate sample must be obtained for each parameter of interest. Samples at the same sampling point for different parameters should be collected from adjacent 100 cm<sup>2</sup> areas (i.e., the same area should not be swabbed with several solvents or twice with the same solvent).

f. Viton **gloves** are recommended to protect project personnel from the sampling solvents and to provide the necessary flexibility to perform the sampling. Sampling supplies should be checked early to ensure these gloves are in stock. The use of forceps or tongs is also recommended to avoid direct contact with solvents. Gloves should be changed immediately if contamination occurs.

g. Sample blanks must be collected with all wipe samples to address matrix interference and collection material/solvent contamination. Wet blanks consist of the collection material wetted with solvent at the sample site and placed directly into a sample vial. Wet blanks must be collected with every sampling project, one blank per every ten wipe samples. Dry blanks are simply unwetted collection material placed into the sample vial at the sampling site. Dry blanks should be collected if contaminants of interest are similar in nature to the extraction solvent used. The necessity of dry blanks should be determined on a project by project basis. Sample blanks may be requested by each individual branch performing laboratory analysis. For example, the CAB demands that a wet blank be submitted with each sample set to ascertain the concentration of nitrobenzenes and interferences created by the acetone/nitrobenzene.

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Table 6-1  
Parameters and Appropriate Solvents

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| Parameter                      | Solvent                               | Remarks*                                   |
|--------------------------------|---------------------------------------|--|
| Metals                         | 1 0 <sup>-3</sup> N Nitric Acid       | MAB, RICD                                  |
| PCBs                           | Hexane                                | PAB, OECD                                  |
| Explosives                     | Acetonitrile                          | SAB, OECD                                  |
| Pesticides                     | Hexane                                | PAB and/or Entomological Sciences Division |
| Herbicides                     | Hexane                                | PAB, OECD                                  |
| Semivolatile Organic Compounds | 70% Methylene Chloride<br>30% Acetone | SAB, OECD                                  |
| Volatile Organic Compounds     | Procedure not Recommended             |  |

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\*Contact the appropriate laboratory for further guidance

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**6-8. SAMPLE MANAGEMENT/FIELD DECONTAMINATION**

a. Guidance on sample handling, chain-of-custody, and documentation may be found in Chapter 3.

b. The requirements listed in Table 6-2 must be adhered to for appropriate preservations, holding times, containers, and quantity requirements.

c. The shipping of waste samples may become more involved, especially when dealing with a pure waste as opposed to a solid or liquid containing some concentration Of the waste.

d. The distinction must be made between environmental samples and **hazardous material (HM)/HW** samples. The EPA has offered some guidance for the packing, parking, labeling, and shipping of these samples. This guidance is contained in Appendix D.

e. In the event that field decontamination of sampling equipment is required, the procedures which should be followed are those described in paragraph 5-5.

Table 6-2  
Waste Sample Collection Requirements (1,2)

| Analysis   | Minimum Quantity | Container (3)                  | Special Preparation | Preservation | Holding Time                               |
|--|------------------|--------------------------------|---------------------|--------------|--|
| Reactivity (4)   | 2-3 gal          | Plastic Pail                   | None                | Cool (5)     | Not established                            |
| Corrosivity (pH)   | 150 mL           | G, P                           | None                | Cool (5)     | 2 days                                     |
| Ignitability (Flashpoint)                                  | 500 mL           | G, Teflon Lid                  | None                | Cool (5)     | Not established                            |
| TCLP Toxicity (Metals)                                     | 500 g            | Wide Mouth                     | None                | Cool (5)     | Not established                            |
| TCLP (Pesticides and Herbicides) Semivolatiles & Volatiles |                  | 1.0-L Wide Mouth G, Teflon Lid | None                | Cool (5)     | 14 days - extraction<br>30 days - analysis |
| Bulk Identification  | 40 g (x2)        | G, Teflon Lid                  | None                | Cool (5)     | Not established                            |

**NOTES:**

- (1) This table contains information for the collection of waste samples for purposes of identifying HW characteristics or hazardous constituents (Bulk identification **analysis**). If other analyses are desired, follow requirements listed in Table 5-1 - Soil Sample Collection Requirements.
- (2) If it is suspected that samples exhibit one or more HW characteristic(s), great care must be taken during handling and transport. Personal protection may be required. See Appendix D for information regarding sample shipping. See Appendix A, reference 31 for health and safety information.
- (3) P = polyethylene, G = glass.
- (4) This analysis is not currently performed by USAEHA laboratories or laboratories under contract to USAEHA. Samples may be sent to the U.S. Bureau of Mines for analysis.
- (5) It is not required that samples be preserved by cooling to 4 °C. However, it is generally recommended that samples be kept cool to prevent reactions from occurring during handling and transport.

**CHAPTER 7  
AIR POLLUTION SAMPLING/ANALYSIS**

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**7-1. AMBIENT AIR SAMPLING**

a. **Definition:** Sampling of outside air not contained in a stack or duct.

b. **POC:** Thomas Guinivan, P.E.  
Chief, Air Quality Management Branch  
(410) 671-3500/3954/2509  
DSN: 584-/3954/2509

**7-2. POINT SOURCE SAMPLING**

a. **Definition:** Sampling of air contained in a duct, vent, or exhaust stack from a specific process such as a incinerator, boiler, or manufacturing process.

b. **POC:** David Daughdrill  
Chief, Air Pollution Management Branch  
(410) 671-3500/3954/2509  
DSN: 584-3500/3954/2509

**7-3. INDOOR AIR POLLUTION**

a. **Definition:** Air in which there are no known contaminants at harmful concentrations as determined by cognizant authorities and with which a substantial majority (80 percent or more) of the people exposed do not express dissatisfaction.

b. **POC:** Steve Graham  
Chief, Field Services Branch  
Industrial Hygiene Division  
(410) 671-2559  
DSN: 584-2559

## CHAPTER 8 SAMPLE ANALYSIS AND DATA REPORTING

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### 8-1. INTRODUCTION

a. To achieve valid analytical results, follow the sample collection procedures and QA/QC measures in this TG.

(1) Once a sample has been collected, transported to USAEHA, processed through AQAD, and delivered to the appropriate laboratory branch or contract laboratory, it is to be analyzed. However, the project officer's responsibility does not end when the samples reach the laboratory.

(2) This TG does not attempt to discuss specific laboratory methods or protocols; however, it should be stressed that sample analysis is an intrinsic part of the sampling project.

b. This chapter discusses some considerations affecting sample analysis, and ways to promote timely and accurate reporting of results through coordination with the laboratories and AQAD.

### 8-2. ANALYTICAL METHODS

a. Most methods used for routine analyses have been developed and approved by the EPA. Most of these methods are listed in two major publications--

(1) SW-846 (Test Methods for Evaluating Solid Waste, Volume 1 A: Laboratory Manual Physical/Chemical Methods).

(2) 40 CFR 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

Included in these manuals are analyses for metals, inorganic nonmetals, physical properties, organics (including purgeable and extractable organic compounds, pesticides, and PCBs), and HW characteristics (ignitability, corrosivity, reactivity, and toxicity characteristic leaching procedure (TCLP)).

b. In some cases, more than one acceptable method of extraction and analysis exists for a particular analyte.

(1) The project officer(s)--

(a) Should always contact the laboratory chemist and AQAD chemist, if their needs require the use of a certain method, or if they do not understand the differences between the methods.

(b) May also specify a particular method on either the original notification or the memorandum for record (MFR) that accompanies a sample.

(2) Table 8-1 lists the routine analytes with the corresponding EPA-approved methods. Table 8-1 is shown on pages 8-4 through 8-8.

(3) Table 8-2 is a complete list of the compounds classified as priority pollutants, Table 8-2 is shown on pages 8-9 through 8-12.

c. Several factors may determine which analytical method is to be used.

(1) The nature of the sample is one example. Samples with extreme or unusual physical properties, or ones containing various constituents or impurities, may interfere with the application of some procedures.

(2) The desired detection limit is another factor that influences selection of an analytical method. Detection limits are discussed in paragraph 8-3.

d. There may be instances when analyses for unusual or atypical analytes are required, especially those normally performed by liquid chromatograph (LC) or gas chromatograph-mass spectrometry (GC-MS) techniques. Examples are breakdown products of munitions-related compounds.

(1) In these cases, the individual laboratory will require additional time to develop methodology, because the acquisition of standard solutions of the compounds in question may be necessary.

(2) Check with the laboratory well in advance of Submitting analytical requests for such services.

### 8-3. DETECTION LIMITS

a. Certain detection limits are routinely achieved by the laboratories. A list of these routine levels for each analyte may be found in Table 8-1.

(1) Special circumstances, such as the character of the sample, may make the routine levels unattainable.

(2) In other cases, State regulatory requirements may require detection limits that are lower than usual. It is important that the project officer be knowledgeable of State requirements and communicate those requirements to the laboratory chemist. The sample may then be prepared according to methods that will achieve the desired detection limit if possible.

b. A parameter which poses a problem concerning the level of detection is dioxin. Analyses for this parameter is not routinely performed. Due to its toxic nature, the tests for dioxin are screening analyses. If the screening procedure indicates the presence of dioxin, more specific tests are required.

#### 8-4. DATA REPORTING

a. General. Appendix F details the responsibilities of the project officer in requesting analytical support and submitting **samples for analysis**. These procedures must be closely followed to facilitate proper sample management.

(1) **Once samples have been** received by AQAD, they **are** assigned 5-character alphanumeric "AQAD numbers." These numbers must be referenced when requesting information about the samples either from AQAD or the individual laboratories.

(2) The AQAD officially tracks samples from their submittal until results are reported.

b. **Sample coordinator.** Each division submitting samples on a regular basis has one person designated as sample coordinator. The sample coordinator assists in submitting the proper paperwork and tracks the samples.

c. **Receipt of results.** Following a review by the appropriate laboratory and/or AQAD, completed analytical data is then reported to the division sample coordinator who transfers it to the project officer.

(1) All analytical results for QC samples (whether prepared in the field or laboratory) should be reported at this time.

(2) A project officer should always notify the sample coordinator when all results for a project have been completed.

d. **Questions concerning data.** When making inquires, the project officer **must** reference the AQAD sample numbers or the project job number.

(1) Questions regarding sample status should be referred to AQAD.

(2) Questions concerning data interpretation should be discussed directly with the laboratory which performed the analyses (see analytical report for laboratory identification).

#### 8-5. HAZARDOUS WASTE SAMPLE DISPOSAL

Samples which **have** been analyzed and are to be discarded must be disposed of **according to** RCRA regulations. These regulations, contained in 40 CFR 260 through 267, 270, and 271, govern the disposal of **HWs**.

a. Grossly contaminated samples or those originating from HW sites should also be suspected of meeting HW criteria.

b. The Hazardous and Medical Waste Branch, Waste Disposal Engineering Division (**WDED**), should be consulted in determining the status of questionable samples.

c. Water and soil samples collected for routine monitoring will not usually fall into this category.

Table 8-1  
Typical Analytical Methods and Detection Limits for Routine Analytes

| Parameter Group       | Analyte(s)       | Medium       | Analytical Method Method No. Reference or Method Description(I) | Method Reference(B) | Typical Detection Limit |
|-----------------------|------------------|--------------|---|---------------------|-------------------------|
| Metals (4)            | Ag               | Water        | 7760, 6010  | SW                  | 0.025 mg/L              |
|                       | Ag               | Water        | 7761  | SW                  | 0.0010 mg/L             |
|                       | AS               | Water        | 7060, 7061  | SW                  | 0.0050 mg/L             |
|                       | Ba               | Water        | 7080  | SW                  | 0.30 mg/L               |
|                       |                  | Water        | 7081  | SW                  | 0.050 mg/L              |
|                       |                  | Water        | 6010  | SW                  | 0.010 mg/L              |
|                       | Be               | Water        | (2)   |                     | 0.0010-0.050 mg/L       |
|                       | Cd               | Water        | 7130, 6010  | SW                  | 0.010 mg/L              |
|                       |                  | Water        | 7131  | SW                  | 0.0005 mg/L             |
|                       | Cr               | Water        | 7190, 6010  | SW                  | 0.025 mg/L              |
|                       |                  | Water        | 7191  | SW                  | 0.0010 mg/L             |
|                       | Cr <sup>+6</sup> | Water        | 7195/7190   | SW                  | 0.025 mg/L              |
|                       |                  | Water        | 7195/7191   | SW                  | 0.0010 mg/L             |
|                       |                  | Water        | 6010  | SW                  | 0.025 mg/L              |
|                       | CU               | Water        | (2)   |                     | 0.0010-0.025 mg/L       |
|                       | Fe               | Water        | (2)   |                     | 0.020-0.10 mg/L         |
|                       | Hg               | Water        | 7471  | SW                  | 0.0002 mg/L             |
|                       | K                | Water        | (2)   |                     | 0.50 mg/L               |
|                       | Mg               | Water        | (2)   |                     | 0.50 mg/L               |
|                       | Mn               | Water        | (2)   |                     | 0.0010-0.030 mg/L       |
|                       | Na               | Water        | (2)   |                     | 1.0 mg/L                |
|                       | Ni               | Water        | 7520, 6010  | SW                  | 0.10 mg/L               |
|                       |                  | Water        | 7521  | SW                  | 0.010 mg/L              |
|                       | Pb               | Water        | 7420  | SW                  | 0.20 mg/L               |
|                       |                  | Water        | 7421  | SW                  | 0.0010 mg/L             |
|                       |                  | Water        | 6010  | SW                  | 0.50 mg/L               |
|                       | Sb               | Water        | 7040  | SW                  | 0.50 mg/L               |
| Water                 |                  | 6010         | SW  | 0.10 mg/L           |                         |
| Se                    | Water            | 7740, 7741   | SW  | 0.0010 mg/L         |                         |
| Tl                    | Water            | (2)          |   | 0.0010-1.0 mg/L     |                         |
| Metals, TCLP (4)      | Zn               | Water        | (2)   |                     | 0.015 mg/L              |
|                       | AS               | Soil/Sludges | 1311 (5)  | SW                  | 0.5 mg/L                |
|                       | Ag               | Soil/Sludges | 1311 (5)  | SW                  | 0.5 mg/L                |
|                       | Ba               | Soil/Sludges | 1311 (5)  | SW                  | 10.0 mg/L               |
|                       | Cd               | Soil/Sludges | 1311 (5)  | SW                  | 0.1 mg/L                |
|                       | Cr               | Soil/Sludges | 1311 (5)  | SW                  | 0.5 mg/L                |
|                       | Hg               | Soil/Sludges | 1311 (5)  | SW                  | 0.02 mg/L               |
|                       | Pb               | Soil/Sludges | 1311 (5)  | SW                  | 0.5 mg/L                |
| Se                    | Soil/Sludges     | 1311 (5)     | SW  | 0.1 mg/L            |                         |
| Organics-Extractables | Acid/Base        | Water        | 625   | MO                  | 0.01 mg/L (9)           |
|                       | Neutrals         | Water        | 3510/8270   | SW                  | 0.001-0.01 mg/L (9)     |
|                       |                  | Soil         | 3550/8250, 8270   | SW                  | 0.3 mg/kg               |
|                       | Volatile6        | Water        | 624 (10)  | MO                  | 0.003 mg/L (9)          |
|                       |                  | Water        | 5030/8260 (11)  | SW                  | 0.002-0.01 mg/L (9)     |
|                       |                  | Soil         | 5030/8260 (11)  | SW                  | 1.0 ppm (9)             |
|                       | Water            | 5242         |   | 0.0005-10 mg/L      |                         |

See Notes on pages 8-7 and 8-8.

Table 8-1  
 Typical Analytical Methods and Detection Limits for Routine Analytes (Continued)

| Parameter Group              | Analyte(s)                     | Medium                 | Analytical Method Method No. Reference or Method Description(1) | Method Reference(3) | Typical Detection Limit |
|------------------------------|--------------------------------|------------------------|---|---------------------|-------------------------|
| Organics-Explosives (12)     | HMX                            | Water                  | HPLC  | AC                  | 100. ppb                |
|                              | RDX                            | Water                  | HPLC  | AC                  | 30. ppb                 |
|                              | 2,4-DNT                        | Water                  | GC  | JC                  | 1. ppb                  |
|                              | 2,6-DNT                        | Water                  | GC  | JC                  | 1. ppb                  |
|                              | TNT                            | Water                  | GC  | JC                  | 1. ppb                  |
|                              | Tetryl                         | Water                  | GC  | JC                  | 10. ppb                 |
|                              | TNB                            | Water                  | GC  | JC                  | 1. ppb                  |
|                              | HMX                            | Soil                   | HPLC  | AJ                  | 1. ppm                  |
|                              | RDX                            | Soil                   | HPLC  | AJ                  | 1. ppm                  |
|                              | 2,4-DNT                        | Soil                   | HPLC  | AJ                  | 1. ppm                  |
|                              | 2,6-DNT                        | Soil                   | HPLC  | AJ                  | 1. ppm                  |
|                              | TNT                            | Soil                   | HPLC  | AJ                  | 1. ppm                  |
|                              | Tetryl                         | Soil                   | HPLC  | AJ                  | 5. ppm                  |
|                              | TNB                            | Soil                   | HPLC  | AJ                  | 1. ppm                  |
| Organics-Fuels               | JP-4                           | Water                  | 503   | SM                  | 1. ppm                  |
|                              | Gasoline                       | Water                  | 503   | SM                  | 1. ppm                  |
|                              | Fuel Oil                       | Water                  | 503   | SM                  | 1. ppm                  |
|                              | #2 Kerosene                    | Water                  | 503   | SM                  | 1. ppm                  |
| Total Petroleum-Hydrocarbons | Total Petroleum-Hydrocarbons   | Water & Soil           | 418.1   | MW                  | 1. mg/L                 |
|                              |                                | Water & Soil           | GC  | (2)                 | 0.1 mg/L                |
| Organics-Pesticides          | Pesticides (7)                 | Potable Water          |   | MP                  | <0.001 mg/L (6)         |
|                              | Pesticides (7)                 | Effluent, Ground Water | 608   | MO                  | <0.001 mg/L (8)         |
|                              | Pesticides/PCBs Herbicides     | Soil                   |   | PM                  | <1. ppm (9)             |
| Organics TCLP                | Benzene                        | Soil                   | 1311 (5)  | SW                  | 0.07 mg/L               |
|                              | Carbontetra-chloride           | Soil                   | 1311 (5)  | SW                  | 0.07 mg/L               |
|                              | Chlordane                      | Soil                   | 1311 (5)  | SW                  | 0.03 mg/L               |
|                              | Chlorobentene                  | Soil                   | 1311 (5)  | SW                  | 1.4 mg/L                |
|                              | Chloroform                     | Soil                   | 1311 (5)  | SW                  | 0.07 mg/L               |
|                              | o-Cresol                       | Soil                   | 1311 (5)  | SW                  | 10.0 mg/L               |
|                              | m-Cresol                       | Soil                   | 1311 (5)  | SW                  | 10.0 mg/L               |
|                              | p-Cresol                       | Soil                   | 1311 (5)  | SW                  | 10.0 mg/L               |
|                              | 24-D                           | Soil                   | 1311 (5)  | SW                  | 1.4 mg/L                |
|                              | 1,4-Dichlorobenzene            | Soil                   | 1311 (5)  | SW                  | 10.8 mg/L               |
|                              | 1,2-Dichloroethane             | Soil                   | 1311 (5)  | SW                  | 4.3 mg/L                |
|                              | 1,1-Dichloroethylene           | Soil                   | 1311 (5)  | SW                  | 0.1 mg/L                |
|                              | 2,4-DNT                        | Soil                   | 1311 (5)  | SW                  | 0.13 mg/L               |
|                              | Endrin                         | Soil                   | 1311 (5)  | SW                  | 0.003 mg/L              |
|                              | Heptachlor (and its hydroxide) | Soil                   | 1311 (5)  | SW                  | 0.001 mg/L              |
|                              | Hexachlorobenzene              | Soil                   | 1311 (5)  | SW                  | 0.13 mg/L               |

See Notes on pages 8-7 and 8-8.

Table 8-1  
Typical Analytical Methods and Detection Limits for Routine Analytes (Continued)

| Parameter Group | Analyte(s)                       | Medium | Analytical Method Method No. Reference or Method Description(1) | Method Reference(3) | Typical Detection Limit |
|-----------------|----------------------------------|--------|---|---------------------|-------------------------|
|                 | Hexachloro-1,3-butadiene         | Soil   | 1311 (5)  | SW                  | 0.72 mg/L               |
|                 | Hexachloroethane                 | Soil   | 1311 (5)  | SW                  | 4.3 mg/L                |
|                 | Lindane                          | Soil   | 1311 (51)   | SW                  | 0.06 mg/L               |
|                 | Methoxychlor                     | Soil   | 1311 (5)  | SW                  | 1.4 mg/L                |
|                 | Methyl Ethyl Ketone              | Soil   | 1311 (5)  | SW                  | 7.2 mg/L                |
|                 | Nitrobenzene                     | Soil   | 1311 (5)  | SW                  | 0.13 mg/L               |
|                 | Pentachlorophenol                | Soil   | 1311 (5)  | SW                  | 3.6 mg/L                |
|                 | Pyridine                         | Soil   | 1311 (5)  | SW                  | 5.0 mg/L                |
|                 | Tetrachloroethylene              | Soil   | 1311 (5)  | SW                  | 0.1 mg/L                |
|                 | Toxaphene                        | Soil   | 1311 (5)  | SW                  | 0.07 mg/L               |
|                 | Trichloroethylene                | Soil   | 1311 (5)  | SW                  | 0.07 mg/L               |
|                 | 2,4,5-Trichlorophenol            | Soil   | 1311 (5)  | SW                  | 5.8 mg/L                |
|                 | 2,4,6-Trichlorophenol            | Soil   | 1311 (5)  | SW                  | 0.30 mg/L               |
|                 | 2,4,5-TP (Silvex)                | Soil   | 1311 (5)  | SW                  | 0.14 mg/L               |
|                 | Vinyl Chloride                   | Soil   | 1311 (5)  | SW                  | 0.05 mg/L               |
| Organics        | TOX                              | Water  | (2)   |                     | 10. ppb                 |
| Inorganic       | Acidity                          | Water  | (2)   |                     | 2. mg/L                 |
| Nonmetals (4)   | T-ALK                            | Water  | (2)   |                     | 2. mg/L                 |
|                 | NH <sup>3</sup> /N               | Water  | (2)   |                     | 0.1 mg/L N              |
|                 | BOD                              | Water  | (2)   |                     | 1.0 mg/L                |
|                 | Bromide                          | Water  | (2)   |                     | 2.0 mg/L Br             |
|                 | COD                              | Water  | (2)   |                     | 25. mg/L                |
|                 | Cl                               | Water  | (2)   |                     | 1. mg/L                 |
|                 | Chlorine                         | Water  | (2)   |                     | 0.05 mg/L               |
|                 | COND                             | Water  | (2)   |                     | 0.10 μmhos/cm           |
|                 | CN                               | Water  | (2)   |                     | 0.01 mg/L               |
|                 | Fluoride                         | Water  | (2)   |                     | 0.20 mg/L               |
|                 | Iodide                           | Water  | (2)   |                     | 2.0 mg/L                |
|                 | NO <sub>2</sub> /NO <sub>3</sub> | Water  | (2)   |                     | 0.05 mg/L N             |
|                 | NO <sub>2</sub> /N               | Water  | (2)   |                     | 0.05 mg/L N             |
|                 | NO <sub>3</sub> /N               | Water  | (2)   |                     | 0.05 mg/L N             |
|                 | Oil & Grease                     | Water  | (2)   |                     | 1.0mg/L                 |
|                 | pH                               | Water  | (2)   |                     | 0.1 pH                  |
|                 | Phenols                          | Water  | (2)   |                     | 0.01 mg/L               |
|                 | Phosphate, total                 | Water  | (2)   |                     | 0.02 mg/L               |
|                 | Phosphate, ortho                 | Water  | (2)   |                     | 0.02 mg/L               |
|                 | TDS                              | Water  | (2)   |                     | 1.0 mg/L                |
|                 | TVDS                             | Water  | (2)   |                     | 1.0mg/L                 |
|                 | TS                               | Water  | (2)   |                     | 1.0mg/L                 |
|                 | TVS                              | Water  | (2)   |                     | 1.0mg/L                 |
|                 | TSS                              | Water  | (2)   |                     | 1.0 mg/L                |
|                 | TVSS                             | Water  | (2)   |                     | 1.0 mg/L                |
|                 | Sulfide                          | Water  | (2)   |                     | 0.1 mg/L                |
|                 | Sulfate                          | Water  | (2)   |                     | 1. mg/L                 |
|                 | TKN                              | Water  | (2)   |                     | 0.20 mg/L               |
|                 | TOC                              | Water  | (2)   |                     | 0.5 mg/L                |
|                 | Turbidity (TURB)                 | Water  | (2)   |                     | 0.20 NTU                |

See Notes on pages 8-7 and 8-8.

Table 8-1  
 Typical Analytical Methods and Detection Limits for Routine Analytes (Continued)

| Parameter Group | Analyte(s)                    | Medium | Analytical Method Method No. Reference or Method Description(1) | Method Reference(3) | Typical Detection Limit |
|-----------------|-------------------------------|--------|---|---------------------|-------------------------|
| Radiochemistry  | Gross alpha                   | Water  | (2)   |                     | 2. pCi/L                |
|                 | Gross beta                    | Water  | (2)   |                     | 2. pCi/L                |
|                 | Tritium                       | Water  | (2)   |                     | 600. pCi/L              |
|                 | Strontium 90                  | Water  | (2)   |                     | 1. pCi/L                |
|                 | Uranium                       | Water  | (2)   |                     | 2. µg/L                 |
|                 | Radium 226                    | Water  | (2)   |                     | 0.2 pCi/L               |
|                 | Radium 228                    | Water  | (2)   |                     | 1. pCi/L                |
|                 | Photon Emitters by Gamma Spec | Water  | (2)   |                     | 10. A pCi/L*            |
|                 | Radon 222                     | Water  | (2)   |                     | 20. pCi/L               |

\* Where A is the abundance value, the photons per nuclear transformation of the most abundant photon with energy greater than 100 keV.

NOTES:

- (1) Two methods listed with a comma indicate two alternative methods. Methods listed with a slash (/) indicate that both methods are used in succession.
- (2) No standard method or several acceptable methods exist for this analyte. A laboratory chemist must be consulted if specific detection units or methods are desired.
- (3) Method Reference Codes are as follows:

AC = Anal Chem 58:170 (1986), Anal Chem 58:176 (1986).  
 AJ = AIHA Journal 45:22-6 (1984).  
 JC = Journal of Chrom Sci 23:532-4 (1985).  
 MC = Method for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents. National Pollutant Discharge Elimination System, Appendix A, Federal Register 38, No. 75, Part II.  
 MO = Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, US EPA, 40 CFR 136, Appendix A.  
 MP = Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water, US EPA. Cincinnati, Ohio, 45258, July 1978. (NOTE: Some modifications are made to this method by USAEHA laboratories.)  
 MW = EPA Method for Chemical Analysis of Water and Wastes, EPA Document No. 600/4-79-020 (Method 413.2), Revised Mar 83.  
 PM = Pesticide Monitoring Special Study No. 44-0131-77, Pesticide Recovery Studies for Evaluation of Department of the Army Pesticide Monitoring Program Soil and Sediment Analysis Methodology, Part I, Determination of Pesticide and Polychlorinated Biphenyl Recoveries from Soil Extracted Immediately Following Fortification, October-December 1976: ADA035782.  
 SM = Standard Methods for the Examination of Water and Wastewater, 17th Edition (1989).  
 SW = SW-846, Test Methods for Evaluating Solid Waste, Volume 1 A: Laboratory Manual Physical/Chemical Methods, US EPA, revised 3d Edition, 1990.

- (4) Solid samples will have varying detection limits and units depending upon sample size and matrix interference. The detection limits listed are those Metals Analysis Branch (MAB) and NMAB generally to achieve.
- (5) Method 131.1 is the TCLP. This procedure would be followed by the applicable method in SW-846 for detection of the specific analyte(s).
- (6) Detection limits vary with each analyte. The exceptions which exceed 0.001 mg/L are toxaphene and malathion, with detection limits of 0.0016 mg/L.
- (7) Also included are herbicides and PCBs.

## NOTES (Continued):

- (8) Detection limits vary with each *analyte*. Exceptions which *exceed* 0.001 mg/L are technical chlordane (0.0012 mg/L), methoxychlor (0.0016 mg/L), toxaphene (0.0016 mg/L), and malathion (0.0016 mg/L).
- (9) Detection limits vary with each *analyte*.
- (10) An alternative to EPA Method 624 is the use of EPA 601 or 602, which are designed specifically for the detection of halogenated VOCs or aromatic VOCs, respectively. These methods should be requested only when a project requires the detection of targeted compounds at lower detection limits.
- (11) An alternative to SW-846 Method 8260 is the use of Methods 8010 or 8020, which are designed specifically for the detection of halogenated VOCs or aromatic VOCs, respectively. These methods should be requested only when a project requires the detection of targeted compounds at lower detection limits.
- (12) Lower detection limits can be obtained on drinking water sample with prior notification of the Chromatographic Analysis Branch (CAB) personnel.

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Table 8-2  
List of Priority Pollutants

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**1. METALS (13)**

Antimony Sb  
Arsenic As  
Beryllium Be  
Cadmium Cd  
Chromium Cr  
Copper Cu  
Lead Pb  
Mercury Hg  
Nickel Ni  
Selenium Se  
Silver Ag  
Thallium Tl  
Zinc Zn

**2. VOLATILE ORGANIC COMPOUNDS (60)**

Benzene  
Bromobenzene  
Bromochloromethane  
Bromodichloromethane  
Bromoform  
Bromomethane  
N-butylbenzene  
n-Butylbenzene  
tert-Butylbenzene  
Carbon tetrachloride  
Chlorobenzene  
Chloroethane  
Chloroform  
Chloromethane  
2-Chlorotoluene  
4-Chlorotoluene  
Dibromochloromethane  
1,2-dibromo-3-chloropropane  
1,2-dibromoethane  
Dibromomethane  
1,2-dichlorobenzene  
1,3-dichlorobenzene  
1,4-dichlorobenzene  
Dichlorodifluoromethane  
1,1-dichloroethane  
1,2-dichloroethane  
1,1-dichloroethylene  
cis-1,2-dichloroethylene

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Table 8-2  
List of Priority Pollutants (Continued)

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## 2. VOLATILE ORGANIC COMPOUNDS (60) (Continued)

trans-1,2-dichloroethylene  
1,2-dichloropropane  
1,3-dichloropropane  
2,2-dichloropropane  
1,1-dichloropropylene  
cis-1,3-dichloropropylene (cis-1,3-dichloropropene)  
trans-1,3-dichloropropylene (trans-1,3-dichloropropene)  
Ethylbenzene  
Hexachlorobutadiene  
Isopropylbenzene  
4-isopropyltoluene  
Methylene chloride  
Naphthalene  
N-propylbenzene  
Styrene  
1,1,1,2-tetrachloroethane  
1,1,2,2-tetrachloroethane  
Tetrachloroethylene  
Toluene  
1,2,3-trichlorobenzene  
1,2,4-trichlorobenzene  
1,1,1-trichloroethane  
1,1,2-trichloroethane  
Trichloroethylene  
Trichlorofluoromethane  
1,2,3-trichloropropane  
1,2,4-trimethylbenzene  
1,3,5-trimethylbenzene  
Vinyl chloride  
o-Xylene  
m-Xylene  
p-Xylene

## 3. BASE-NEUTRAL EXTRACTABLE ORGANIC COMPOUNDS (46)

Acenaphthene  
Benzidine  
1,2,4-trichlorobenzene  
Hexachlorobenzene  
Hexachloroethane  
Bis(2-chloroethyl) ether  
2-Chloronaphthalene  
1,2-dichlorobenzene  
1,3-dichlorobenzene

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Table 8-2  
List of Priority Pollutants (Continued)

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**3. BASE-NEUTRAL EXTRACTABLE ORGANIC COMPOUNDS (46) (Continued)**

1,4-dichlorobenzene  
3,3-dichlorobenzidine  
2,4-dinitrotoluene  
2,6-dinitrotoluene  
1,2-diphenylhydrazine  
Fluoranthene  
4-chlorophenyl phenyl ether  
4-bromophenyl phenyl ether  
Bis(2-chloroisopropyl) ether  
Bis(2-chloroethoxy) methane  
Hexachlorobutadiene  
Hexachlorocyclopentadiene  
Isophorone  
Naphthalene  
Nitrobenzene  
N-nitrosodimethylamine  
N-nitrosodiphenylamine  
N-nitrosodi-n-propylamine  
Butyl benzyl phthalate  
Di-n-butyl phthalate  
Di-n-octyl phthalate  
Diethyl phthalate  
Dimethyl phthalate  
Benzo(a)anthracene (1,2-benzanthracene)  
Benzo(a)pyrene (3,4-benzopyrene)  
3,4-benzofluoranthene  
Benzo(k)fluoranthene (1,12-benzofluoranthene)  
Chrysene  
Acenaphthylene  
Anthracene  
Benzo(ghi)perylene (1,12-benzoperylene)  
Fluorene  
Phenanthrene  
Dibenzo(a,h)anthracene (1,2:5,6-dibenzanthracene)  
Indeno(1,2,3-cd)pyrene  
Pyrene  
Bis(2-ethylhexyl) phthalate

**4. ACID EXTRACTABLE ORGANIC COMPOUNDS (11)**

2,4,6-Trichlorophenol  
Parachlorometacresol  
2-chlorophenol  
2-nitrophenol

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Table 8-2  
List of Priority Pollutants (Continued)

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**4. ACID EXTRACTABLE ORGANIC COMPOUNDS (11) (Continued)**

Pentachlorophenol  
2,4-dimethyphenol  
4-Nitrophenol  
2,4-dinitrophenol  
4,6-dinitro-o-cresol  
2,4-dichlorophenol  
Phenol

**5. PESTICIDES AND PCBs (26)**

Aldrin  
Dieldrin  
Chlordane  
4,4'-DDT  
4,4'-DDE  
4,4'-DDD  
alpha-Endosulfan  
beta-Endosulfan  
Endosulfan sulfate  
Endrin  
Endrin aldehyde  
Heptachlor  
Heptachlor epoxide  
alpha-BHC  
beta-BHC  
gamma-BHC (Lindane)  
delta-BHC  
PCB-1242 (Arochlor 1242)  
PCB-1254 (Arochlor 1254)  
PCB-1221 (Arochlor 1221)  
PCB-1232 (Arochlor 1232)  
PCB-1248 (Arochlor 1248)  
PCB-1260 (Arochlor 1260)  
PCB-1016 (Arochlor 1016)  
Toxaphene  
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)

**6. MISCELLANEOUS (2)**

Asbestos  
Total cyanides

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**APPENDIX A**

**REFERENCES**

**SECTION I - PUBLICATIONS**

1. Title 29, CFR, latest rev, Part 1910, Occupational Safety and Health Standards.
2. Title 40, CFR, latest rev, Part 122, EPA Administered Permit Programs: National Pollutant Discharge Elimination System.
3. Title 40, CFR, latest rev, Part 124, Procedures for Decision Making.
4. Title 40, CFR, latest rev, Part 125, Criteria and Standards for the National Pollutant Discharge Elimination System.
5. Title 40, CFR, latest rev, Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants.
6. Title 40, CFR, latest rev, Part 141, National Primary Drinking Water Regulations.
7. Title 40, CFR, latest rev, Part 143, National Secondary Drinking Water Regulations.
8. Title 40, CFR, latest rev, Part 146, Underground Injection Control Program: Criteria and Standards.
9. Title 40, CFR, latest rev, Part 260, Hazardous Waste Management System: General.
10. Title 40, CFR, latest rev, Part 261, Identification and Listing of Hazardous Waste.
11. Title 40, CFR, latest rev, Part 262, Standards Applicable to Generators of Hazardous Waste.
12. Title 40, CFR, latest rev, Part 263, Standards Applicable to Transporters of Hazardous Waste.
13. Title 40, CFR, latest rev, Part 264, Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities.
14. Title 40, CFR, latest rev, Part 265, interim Status Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities.
15. Title 40, CFR, latest rev, Part 266, Standards for the Management of Specific Hazardous Wastes and Specific Types of Hazardous Waste Management Facilities.
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#### SECTION II • FORMS

1. AEHA Label 7 (Used for labeling sample containers).
2. AEHA Form 8-R, Bulk Sample Data.
3. AEHA Form 9-R, Industrial Hygiene Air Sample Data.
4. AEHA Form 75-2, Listing of Specific Chemical Analyses.
5. AEHA Form 235-R, Chain-of-Custody Record.
6. AEHA Form 328-R, Drinking Water Supply Field Data Sheet.
7. AEHA Form 329-R, Sample Alteration Form.
8. AEHA Form 330-R, Request for Laboratory Services.
9. AEHA Form 331-R, Field Corrective Action Form.
10. AEHA Form 332-R, Directorate of Laboratory Sciences Corrective Action Form.

## APPENDIX B

SPECIAL REQUIREMENTS FOR METALS ANALYSES  
IN WASTEWATER, GROUND WATER, AND DRINKING WATER

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**B-1.** Wastewater and ground-water samples for total metals analyses, except mercury and hexavalent chromium, can be sampled as a group. They must be acidified to a pH of <2 with HNO<sub>3</sub>, with the exception of the following wastewater sample types, which should **NEVER** be preserved:

- a. Those which have high specific conductivities (>20,000 micromhos/cm).
- b. Those which are highly alkaline (pH > 11) or acidic (pH < 2).
- c. Those which contain two or more immiscible phases.

E-2. If a project officer desires a parameter not listed in Table 4-1, or detection limits other than those specified, the laboratory branch chief and project chemist should be consulted. All samples with the exception of those for total mercury should be collected in new polyethylene containers. No special rinsing of containers is necessary.

B-3. All samples for total mercury analysis should be collected in new glass containers. They are to be preserved with HNO<sub>3</sub> and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> to give a final sample pH of <2, usually achieved by a final sample concentration of 0.5 percent HNO<sub>3</sub> and 0.05 percent K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>.

B-4. Wastewater and ground-water samples for hexavalent chromium analysis are NOT preserved with chemicals but MUST be kept cold (4 °C) and analyzed within 48 hours of collection. This requirement dictates that the--

- a. Samples arrive at the laboratory no later in the week than Wednesday.
- b. Project chemist be notified of their impending arrival.

B-5. If dissolved metals are required, the fresh sample--

- a. Must be filtered in the field using a 0.45 micron membrane filter
- b. Is then preserved with the appropriate chemicals.

It is not envisioned that filtration would be required when dealing with a potentially hazardous wastewater.

B-6. Historically, the cooled preserved samples have been transported with DOT approval. However, **special** Safety precautions are necessary when handling the chemical preservatives, and care must be taken to avoid contamination of the samples.

APPENDIX C

SPECIAL REQUIREMENTS FOR  
METALS ANALYSES IN SOILS AND SLUDGES

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C-1. The specific reference for analysis of metals in soils and sludges is "Test Methods for Evaluating Solid Waste," SW-846, revised 3d edition. **The** separate methods for parameters of interest are listed in Chapter 3, Section A, Vol. I, of the cited reference.

C-2. Solid samples will have different detection limits and units depending on the sample size analyzed and on matrix interference. Generally, the MAE achieves detection limits an order of magnitude lower than the maximum concentration levels posted in SW-846, section 2.1.4. for EP Toxicity Metals.

C-3. The SW-846 Methods 3010, 3020, **3030**, 3040, **3050**, and 3060 are used for sample preparation; the selection of preparation method depends on the nature of the sample.

a. Toxicity Characteristic Leaching Procedure (TCLP), Method 131 1, the EP for metals toxicity, is provided in SW-846.

b. Method 1330 is the toxic metals extraction procedure for oily wastes.

C-4. Nonliquid samples, whether sediment or solid, are NOT chemically preserved but should be kept cool.

**APPENDIX D**  
**SAMPLE SHIPPING**

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**D-I. REGULATORY REQUIREMENTS - GENERAL**

## a. This TG--

(1) Applies to shipment of samples containing potential HMs or HW's. (Generally, an HW is shipped according to the requirements for the hazard class it falls into as an HM).

(2) Combines good engineering practices with regulatory requirements set forth by DOT.

(3) Has information from EPA/emergency response team (ERT) "Hazardous Materials Incident Response Operations" training course manual (course No. 165.1), and EPA training course "Hazardous Waste Site Sampling" (course No. 161 .5) incorporated.

b. DOT regulations **MUST** be complied with when shipping a known HM. This applies to samples which consist of a pure or nearly pure substance which is hazardous.

(1) Title 49 CFR, parts 171 through 180 contain the HM regulations.

(2) The HM Table (49 CFR 172.101) should be used to identify--

(a) Whether a sample is a listed DOT HM and, if so,

(b) How that sample should be labeled, packaged, marked, and shipped.

c. The DOT regulations specifically address the labeling requirements of packages containing samples under 49 CFR 172.402.

(1) This states that the person responsible for shipment of the sample must make a tentative class assignment based on his knowledge of the material and on the hazard precedence described in 49 CFR 173.2a.

(2) Table D-I provides a prioritized list of the hazard classes, which is used for--

(a) Materials having more than one hazard associated with them, or

(b) In the case of samples, materials for which the hazard is not specifically known.

(3) It is the project officer's responsibility to determine how the samples are to be shipped.

Table D-1  
DOT Hazardous Precedence for HM Shipment

| Category                           | Definition      |
|------------------------------------|-----------------|
| Radioactive material               | 49 CFR 173.403  |
| Poison A                           | 49 CFR 173.326  |
| Flammable gas                      | 49 CFR 173.300  |
| Nonflammable gas                   | 49 CFR 173.300  |
| Flammable liquid                   | 49 CFR 173.115  |
| Oxidizer                           | 49 CFR 173.151  |
| Flammable solid                    | 49 CFR 173.150  |
| Corrosive material (liquid)        | 49 CFR 173.240  |
| Poison B                           | 49 CFR 173.343  |
| Corrosive material (solid)         | 49 CFR 173.240  |
| Irritating material                | 49 CFR 173.381  |
| Combustible liquid (1)             | 49 CFR 173.115  |
| Other regulated material-B (ORM-B) | 49 CFR 173.800  |
| ORM-A                              | 49 CFR 173.605  |
| Combustible liquid (2)             | 49 CFR 173.115  |
| ORM-E                              | 49 CFR 173.1300 |

(1) In containers exceeding 100 gal capacity

(2) In containers having capacities of 110 gal or less

**D-Z. ENVIRONMENTAL SAMPLES AND HM SAMPLES**

**a. Determination of sample type.** The project officer must first determine the type of sample. The EPA has classified sample types as follows:

**(1) Environmental sample.**

(a) In general, environmental samples are not considered grossly contaminated but are taken from a medium (soil or water for example) and contain an undetermined level of hazardous constituents.

(b) Examples are--

1. Soil or residues from **OB/OD** grounds.

- 2. Most drinking water and wastewater samples.
- 3. Soil surrounding drum or pesticide storage sites.

**(2) HM sample.** HM samples--

(a) Are those which are obviously contaminated or possess hazardous characteristics, such as flammability or corrosivity.

**NOTE: DOT criteria for hazardous characteristics differ slightly from those set forth by EPA for defining HW. See Table D-2.**

Table D-2  
Criteria Used to Include Samples Under DOT Regulations

CORROSION - The pH of the sample exceeds the range of 1-13

FLAMMABILITY - The sample has a flash point below 100 °F

POISON - The sample has an anticipated LD50 less than 50 mg/kg

PATHOGENIC TO MAN - The sample causes human disease by external body exposure

THERMAL INSTABILITY - The sample will spontaneously combust

(b) May come from waste drums, storage tanks, dip tanks, or lagoons, for example.

(c) Include battery acids, solvents, caustic cleaners, and some spill residues.

(d) May include soil and water samples if high contamination levels are presumed. Examples of this are heavily contaminated wastewater, or a well where gasoline has leaked from an underground tank, causing the sample to be a flammable liquid.

**(3) Type of sample.** The project officer should use the following questions to help determine the type of sample:

(a) Is the sample the result of a recent spill or leak of a known HM?

(b) Is the soil or water visibly contaminated (e.g., discolored water, heavily saturated soil)?

(c) Is there a strong or irritating odor to the sample?

(d) Would the sample create a hazard if **breakage or leakage occurred during** transportation?

(e) Would the sample create a hazard to laboratory personnel receiving it?

(f) Does the sample contain potentially explosive or reactive constituents in dangerous concentrations?

(g) Does the sample meet any of the criteria in Table D-27

When the answer to any of the preceding questions is yes, the determination would usually be that the sample is a HM sample. If there is substantial doubt as to the sample type, samples should be considered HM samples.

**b. Packaging end shipping of environmental samples.** Environmental samples should be packaged and shipped according to the following procedures.

(1) Place sample container, properly identified and sealed, in a plastic or polyethylene bag, and seal the bag.

(2) Place sample in a fiberboard container or ice chest which has been lined with a large plastic or polyethylene bag.

**NOTE: Some samples require a temperature of 4 °C; therefore, an appropriate container with a freezer pack is recommended. See Tables 4-1, 5-1, or 6-2 to determine whether sample requires refrigeration.**

(3) Pack with enough noncombustible, absorbent, cushioning material to minimize the possibility of the container breaking, and to fully absorb the contents if breakage does occur.

(4) Seal the large bag.

(5) Seal or close the outside container.

(6) The outside of the container should be marked "Environmental Sample." The appropriate side of the container should be marked "This end up" with arrows placed accordingly. No DOT markings or labels are required.

(7) No DOT shipping **papers are required.** There are no DOT restrictions on mode of transportation.

**c. Packaging and shipping of HM samples.**

(1) If the substance in the sample is known or can be identified, the sample must be packaged, marked, labeled, and shipped according to the specific instructions for that material in the DOT HM Table, 49 CFR 172.101.

(a) Note that "limited quantities" exemptions exist for some materials, and this can be applied to sample quantities. See the exception column in the DOT HM Table. For example, if a sample is known to be battery acid, one would see "battery fluid, acid" listed in the DOT HM Table, and then package and ship the sample according to the requirements under 49 CFR 173.244, "Limited quantities of corrosive materials."

(b) If the general hazard class for the sample can be determined based on knowledge of its characteristics, regulations pertaining to that hazard class shall be followed.

1. Hazard classes are listed in the DOT HM Table followed by n.o.s. (not otherwise specified).

2. Table D-2 outlines the criteria used by DOT to determine hazard classes

(2) For samples judged to be HM samples where the material is not listed in the HM Table and the general hazard class is not known, or the sample falls into more than one hazard class, the appropriate hazard class must be chosen from Table D-1. (This list of hazard precedence may also be found in 49 CFR 173.2). This is done by the process of elimination--

(a) Those classes which clearly do not apply may be eliminated.

(b) The first class which can not be ruled out either by knowledge of the material or by testing is the one which dictates the shipping regulations.

(3) Samples which are HW's should be shipped according to the requirements for the corresponding material.

(a) If the word "waste" is not included in the description in the HM Table, the proper shipping name for an HW must include the word "waste." Example: Waste Acetone.

(b) HWs which do not meet the definition of any HM or hazard class must be shipped as ORM-E.

(4) Generally, the first four categories of the hazard precedence list (Radioactive Material, Poison A, Flammable Gas, or Nonflammable Gas) will not apply to samples.

(a) Therefore, unless it can be determined in the field that the sample is not a flammable liquid or solid, the sample should be shipped according to the requirements **for** that category. (The requirements applicable to oxidizers also apply to flammable solids).

(b) The requirements **for packaging** and shipping flammable liquids and solids (meeting the requirements for the "limited quantity" exclusion) are as follows:

1. Collect sample in a container of **16-oz** size or less. (Limited quantities of flammable liquids, for the purpose of the exclusion, are defined as 1 pt or less. For flammable solids, it is defined as 1 pound net weight in inner containers and no greater than 25 pounds net weight in the outer container.) To prevent leakage, fill container so that it would be no more than **90-percent** full at 54 °C.

2. Place sealed container, properly **tagged** and identified, in a **2-mL** thick (or thicker) polyethylene bag, one sample per bag. Position identification tag so it can be read through bag. Seal bag.

3. Place sealed bag inside metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite or diatomaceous earth) between the bottom and sides of the can and bag to prevent breakage and to absorb leakage. Pack one bag per can. Use clips, tape, or other means to hold can lid securely, tightly, and permanently.

4. Place one or more metal cans into a strong outside container, such as a metal ice chest or a DOT-approved fiberboard box. Surround cans with noncombustible, absorbent, cushioning material for stability during transport.

5. Place the following information on the metal cans:

- a. Proper shipping name as found in the HM Table.
- b. Applicable category.
- c. Corresponding United Nations (UN) number.

Example: "Acetone, Flammable Liquid, UN 1090."

6. If the material is not specifically listed in the HM Table, or if its nature is unknown, the label should read: "Flammable Liquid, n.o.s. **UN1993**" or "Flammable Solid, n.o.s. **UN1 325**."

7. Place the following DOT labels on outside of the can or bottle:

- a. "Flammable liquid" or "Flammable solid."
- b. "Dangerous when wet" if material meets definition of water-reactive material.
- c. "Cargo Aircraft **Only**" if net quantity of sample in each outer container is greater than 1 qt or 25 pounds.

8. Place all information on outside shipping container as on can, **specifically--**

- a. Proper shipping name.
- b. UN number.
- c. Applicable labels.
- d. Addressee and addressor.

9. Print "Laboratory Samples" and "This End Up" clearly on top of shipping container.

10. Shipping papers are required. Complete the carrier-provided bill of lading and sign certification statement (if carrier does not provide, use standard industry form). Complete all information as requested.

11. Transport unknown hazardous substance samples classified as flammable by rented or common carrier truck, railroad, or express overnight package services.

a. Do not transport by any passenger-carrying air transport system, even if they have cargo-only aircraft.

(1) The DOT regulations permit regular airline cargo-only aircraft, but **difficulties** with most suggest avoiding them.

(2) Instead, ship by airlines that only carry cargo

b. If transporting by Government-owned vehicle, including aircraft, DOT regulations do not apply.

**APPENDIX E**

**U.S. ARMY ENVIRONMENTAL HYGIENE AGENCY  
CHAIN-OF-CUSTODY**

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**E-1. SCOPE**

This appendix applies to all environmental samples--

- a. Identified as chain-of-custody samples, and
- b. Received at USAEHA and USAEHA direct support activities, as well as contract laboratories which perform analytical services for USAEHA.

**E-2. INTRODUCTION**

The USAEHA conducts environmental monitoring to assure the health and safety of military and civilian personnel located at DA installations within the continental United States (CONUS) and outside the continental United States (OCONUS).

- a. This mission comprises-
  - (1) Preliminary assessment or study of areas of concern by project officers
  - (2) Collection of field samples for measurement of a multitude of chemical parameters.
  - (3) Generation of data from laboratory analyses of these environmental samples
  - (4) Submission of written reports by project officers detailing remedial action required to eliminate the problem(s).
- b. Reports are forwarded to State and/or Federal regulators who determine whether the particular activity is in compliance with applicable laws governing environmental assessment.
- c. In the event of litigation, the processes of sample collection, transferral, analysis, storage, and disposal are documented and comprise a "chain-of-custody" which may be used to trace the possession of sample from the moment of sample collection through analysis to final sample disposal.
- d. Chain-of-custody is, therefore, a critical element of good field and laboratory practices.

**E-3. PURPOSE**

The USAEHA has adopted a policy where chain-of-custody guidelines will be enforced during the sample collection, sample receipt, sample identification, sample analysis, sample storage, and sample disposal processes conducted as part of environmental monitoring and assessment.

a. These actions will--

- (1 ) Establish a reasonable probability that a sample has not been tampered.
- (2) Assure that sample integrity is maintained.
- (3) Assure that possession and handling is documented.
- (4) Assure that this accurate written record will be defensible in court if the necessity arises.

b. In environmental litigation, adherence to chain-of-custody principles has two main goals, to ensure that the sample--

- (1 ) Which is collected is the same sample which is analyzed.
- (2) Is not altered, changed, or tampered between the collection and analysis.

**E-4. CHAIN-OF-CUSTODY GUIDELINES FOR FIELD SAMPLE COLLECTION AND SAMPLE TRANSFER TO LABORATORY**

a. Project officers from the following directorates as well as the direct support activities will be responsible for assuring that proper chain-of-custody protocol is practiced during collection of environmental samples.

- (1) Directorate of Environmental Health Engineering (**DEHE**).
- (2) Directorate of Industrial Hygiene (**DIH**).
- (3) Directorate of Radiation and Entomological Sciences (**DRES**).

b. All actions conducted in the field will be documented in field logbooks as well as on chain-of-custody records. Information which is assigned to each field sample will include the following:

- (1) Source/installation where sample was collected.
- (2) Date and time of collection of field sample.
- (3) Field sample number.

- (4) Analyses desired for sample.
- (5) Project officer who collected sample.
- (6) Project number
- (7) Total number of containers per sample.
- (8) Date of shipment of sample to laboratory.
- (9) Method of shipment (e.g., United Parcel Service (UPS), Federal Express).

c. Upon completion of field sampling, project officers will **carefully** package samples in appropriate transportation containers (coolers, cases, mailers, etc.).

(1) The original chain-of-custody records will be placed in a sealed plastic bag to prevent wetting and sealed inside the respective samples' shipping container.

(2) Transportation containers will then be sealed with tamperproof shipping tape and forwarded to the laboratory for subsequent analyses.

(3) When transferring the "possession" of the container to the next party (i.e., laboratory personnel), the project officer will sign and record the date/time of transfer on the chain-of-custody record included with each group of samples for each transportation container. Figure E-1 shows an example of AEHA Form 235-R (Chain-of-Custody Record). This form will be locally reproduced. A copy for reproduction purposes is located at the back of this TG.

(4) Project officers will retain a photocopy of each chain-of-custody record designated for each transportation container of samples.

d. Transportation containers will be shipped to the laboratory via common carrier (UPS, Federal Express, etc.).

(1) Common carriers will abide by DOT regulations governing shipment of respective sample.

(2) **Upon** receipt of containers from common carrier, chain-of-custody will be relinquished to AQAD.

(3) Any evidence of tampering (e.g., breakage of seal) during shipment by common carrier will be documented upon receipt and inspection of transportation containers by personnel of AQAD.

e. As soon as samples are transferred from AQAD to DLS branch laboratory personnel or contract laboratory personnel, custody will be relinquished to them.



f. If, for any reason, the chain is broken between transfer of samples from field to AQAD or from AQAD to laboratory, a contingency plan will be implemented to--

- (1 ) Determine cause of breakage of chain.
- (2) Perform corrective action to reconstruct chain.

g. A description of the contingency plan is detailed in paragraph E-5b.

#### **E-5. CHAIN-OF-CUSTODY GUIDELINES FOR SAMPLE INPROCESSING, SAMPLE ANALYSIS, AND SAMPLE STORAGE/SAMPLE DISPOSAL AT LABORATORY**

a. Personnel of the DLS will be responsible for implementing procedures which will assure custody of sample from the time of receipt of sample through sample analysis, storage, and finally to sample disposal. These procedures are detailed as follows:

(1) Primary and alternate sample custodians will be designated in writing for each branch and AQAD.

(a) The primary and alternate sample custodians will be--

1. Responsible for receipt and accountability of samples in their respective branch or AQAD.
2. Required to sign chain-of-custody record(s) which accompany samples.
3. Required to transcribe information contained on chain-of-custody records to logbooks for permanent recordkeeping.

(b) A list of primary and alternate sample custodians as well as the branch chief for each branch laboratory will be maintained on file by AQAD. Only these personnel will have access to chain-of-custody sample storage area.

(2) A specific area will be designated for storage of chain-of-custody samples in AQAD until transfer and subsequent storage of samples in DLS branch laboratories.

(a) The AQAD and DLS branch laboratory storage areas will be securely locked.

(b) When custody samples are transferred from AQAD by the AQAD sample custodian to the DLS branch laboratory sample custodian, the person accepting the sample will sign chain-of-custody records indicating transfer of possession of samples to that person.

1. The chain-of-custody records designated for each branch laboratory will specify, via highlighted areas, the sample subsets which will be forwarded to respective branch laboratories.

2. Also, within each branch, when **samples** are transferred to laboratory analysis area, personnel who obtain samples will sign chain-of-custody records indicating transfer of possession to that person until completion of sample analysis at which time the sample and corresponding chain-of-custody records will be returned **to** the designated storage area until sample is disposed.

(3) Access to the laboratory area where custody samples are analyzed will be controlled at all times.

(a) The analyst(s) will ensure that the contents of the sample are not tampered with during the time the sample is in their possession.

(b) A list of authorized personnel who are allowed access to the particular laboratory area will be posted on doors leading to the laboratory area.

(c) Unauthorized personnel are permitted access to laboratory areas containing chain of-custody samples only when escorted by an authorized person.

(4) When entered into the laboratory sample and data management system (i.e., sample tracking and reporting (STARLOG)), the chain-of-custody samples will be flagged as such.

(5) Upon completion of analysis of custody sample(s), the pertinent branch laboratory--

(a) Sample custodian will forward these samples to the AQAD sample custodian for proper storage prior to disposal.

(b) Personnel will forward completed chain-of-custody records for filing in AQAD.

b. A contingency plan will be implemented when the chain is broken (e.g., if samples do not arrive with chain-of-custody records and vice versa, if unauthorized personnel open shipping containers or sign chain-of-custody records upon receipt of samples). This contingency plan will comprise the following steps:

(1 ) Obtain affidavits from all field and laboratory personnel in order to reconstruct the chain.

(a) The affidavit is a sworn statement that the person in question actually relinquished samples to the next individual in the transfer process.

(b) Figure E-2 shows an example of an affidavit,

AFFIDAVIT

State of \_\_\_\_\_ C o u r t y o f \_\_\_\_\_

I do solemnly swear (or affirm) that to the best of my knowledge the environmental samples identified on chain-of-custody record were transferred in a secure manner to the next party indicated on chain-of-custody record. I also solemnly swear (or affirm) that to the best of my knowledge the samples were not damaged or tampered with during process or transfer.

In **WITNESS WHEREOF** I have hereunto set my hand and seal this \_\_\_\_\_ day of \_\_\_\_\_ 19 \_\_\_\_.

Typed Name and Grade/Rank \_\_\_\_\_

Signature and Date \_\_\_\_\_

Subscribed and sworn to (or affirmed) before me this \_\_\_\_\_ day of \_\_\_\_\_

Witness Signature \_\_\_\_\_

My commission expires \_\_\_\_\_

**EXAMPLE**

Figure E-2. Example of an Affidavit

(2) Complete the transfer of custody samples and attach affidavits to chain-of-custody documentation. File information accordingly.

(3) Branch chiefs should review affidavits to ascertain if reconstruction has or has not occurred.

**E-6. SUMMARY**

a. It is essential that a diligent effort be undertaken by representatives of all directorates to achieve, implement, and manage an effective chain-of-custody policy that will be--

(1 ) A model policy for other DA activities.

(2) Impervious to criticism when such information is submitted as evidence in State and Federal enforcement proceedings.

b. This effort will consequently assure maintenance of sample integrity and demonstrate that proper chain-of-custody procedures are being followed.

## APPENDIX F

REQUIREMENTS FOR SUBMITTING SAMPLES TO USAEHA

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## F-I. GENERAL REQUIREMENTS

a. In order to ensure that sufficient and appropriate data is generated to support the objectives of a study, a clear definition of the project needs and requirements should be developed early in the planning process. Laboratory review of such needs and requirements is essential to ensure that all objectives can and will be met.

b. The process of establishing these objectives, often referred to as Data Quality Objectives (DQOs), is presented in Table F-1. Though a less formal approach may be appropriate, **advanced notification to the analytical laboratories is a must!** Generally, individual laboratories should be notified at least 30 days before the expected arrival date of all samples. Special/ emergency situations can be negotiated on a case-by-case basis.

**(1) Initiate laboratory involvement.** Prior to initiating a formal request for **analytical** services, contact the individual laboratory(s). See Table F-2 for a list of the individual laboratory services numbers and the corresponding telephone numbers. Valuable information can be obtained from laboratory personnel by discussing the general purpose and requirements of a study. Discuss special needs/requirements (see F-2. SPECIAL REQUIREMENTS) and confirm the appropriateness of sampling methodologies at this time.

**(2) Complete "Request for Laboratory Services" form.** After receiving verbal confirmation from the individual laboratories, the requestor can complete AEHA Form 330-R (Request for Laboratory Services). Figures F-1 and F-2 show examples of this form and the corresponding instruction sheet. This form should be signed by the program manager or an authorized designee. This form will be locally reproduced. A copy for reproduction purposes is located at the back of this TG.

**(3) Distribute "Request" form.** Copies of the completed form should be forwarded to each of the following:

- (a) AQAD.
- (b) Division Chief(s) of applicable division(s).
- (c) Branch Chief(s) of applicable branch(s).

**(4) Confirm receipt of request.** Before collecting samples, contact the individual laboratory(s) to confirm the receipt and acceptance of the request for services. If possible, obtain the name(s) of the chemist(s) who will be performing the analyses. Changes in the original request or additional information should be provided at this time.

Table F-1  
The Process of **Establishing** Data Quality Objectives (DQOs)

Data Quality Objectives (DQOs) are defined as qualitative and quantitative statements established prior to data collection, which specify the quality of the data needed to support a decision.

The following outline discusses the process of establishing DQOs. Specific tasks required by this process and the parties that are typically responsible for performing these tasks are also described:

| DQO Process  | Specific Tasks  | Responsible Party(s)                          |
|--|---|---|
| <b>STAGE i</b>   |   |   |
| Define the decision types<br><br>[PROJECT OBJECTIVES]  | <ul style="list-style-type: none"> <li>- compile and assess available site/project information</li> <li>- establish purpose of work and define requirements [i.e., to screen, regulatory compliance, or health risk issue?]</li> </ul>  | <p>project officer</p> <p>project officer</p> |
| <b>STAGE ii</b>  |   |   |
| Clarify information needed (sampling and analytical req)<br><br>[BEGIN DEVELOPING SAMPLING PROTOCOL] | <ul style="list-style-type: none"> <li>- provide necessary information to lab with request for analysis (info should include general scope/purpose of project, pertinent regulatory requirements, required analytical methods with detection limits, sample types, sample quantities, and timelines)</li> <li>- confirm understanding of requirements, scope, etc.</li> </ul> | <p>project officer</p> <p>lab personnel</p>   |

\* 'Responsible parties' should be more clearly defined at the onset of a project. Establishing the individuals who will be specifically involved in performing these tasks (such as the lab chemist, AOAD POC, etc.) is an essential part of this DQO process.

Table F - 1  
The Process of Establishing Data Quality Objectives (DQOs) (Continued)

| DQO Process                             | Specific Tasks   | Responsible Party(s)*      |
|---|--|----------------------------|
| <u>STAGE II cont'd</u>                  |  |                            |
| State desired performance level of data | - establish appropriate QA/QC  | both lab & project officer |
| <b>[SAMPLING PLAN]</b>                  | - communicate decisions on appropriate QA/QC with appropriate party  | both lab & project officer |
| <u>STAGE III</u>                        |  |                            |
| Design data collection program/scheme   | - complete sample protocol incorporating the applicable QA/QC methods  | project officer            |
| <b>[FINALIZE SAMPLING PLAN]</b>         | - continue communication, specifically re: sampling methods, timeliness, changes, plan modifications, etc.   | both lab & project officer |
| <u>(STAGE IV)</u>                       |  |                            |
| Review final product                    | - inspect quality of data in regards to: > precision (repeatability)<br>> accuracy (to true value)<br>> representativeness (of actual pop.)<br>> completeness (substantiative?)<br>> comparability (data sets equaliv?)<br>> timeliness (on time?) | both lab & project officer |
| <b>[to determine if DQOs were met]</b>  | determine deficiencies/problem areas and establish a plan of action to address/correct   | both lab & project officer |

3

Table F-2  
 USA/EHA Directorate of Laboratory Services

| Division/Branch   | Telephone Number<br>Commercial/DSN | Analyses  |
|---|------------------------------------|---|
| <b>1 Radiological and Inorganic Chemistry<br/>Division (RICD)</b> | (410) 671-2619/584-2619            |   |
| a. Metals Analysis Branch (MAB)                                   | (410) 671-2637/584-2637            | Total metals, TCLP metals, pH (only with EP metals request)   |
| b. Nonmetals Analysis Branch (NMAB)                               | (410) 671-2637/584-2637            | Inorganic parameters (e.g., ammonia, BOD, COD, chloride, cyanide, grease and oil, hardness, nitrate, TOC, pH, phenol, phosphate, specific conductivity, TDS, TSS, TVS, TVDS, sulfate) |
| c. Radiological Analysis Branch (RAB)                             | (410) 671-2619/584-2619            | Radiochemistry (e.g., gross alpha, gross beta, strontium-90)  |
| <b>2. Organic Environmental Chemistry<br/>Division (OECD)</b>     | (410) 671-3739/594-3739            |   |
| a. Chromatographic Analysis Branch (CAB)                          | (410) 671-2208/584-2208            | Explosives, fuels in water  |
| b. Special Analysis Branch (SAB)                                  | (410) 671-2208/584-2208            | PCBs, Bulk identification, VOCs, acid and base/neutral extractable organics, general organic screen specific organics, flashpoint, TOX.   |
| c. Pesticides Analysis Branch (PAB)                               | (410) 671-2177/584-217-1           | Pesticides, PCBs  |
| <b>3. Analytical Quality Assurance<br/>Division (AQAD)</b>        | (410) 671-3269/584-3269            | Contract analyses.  |

4

REQUEST FOR LABORATORY SERVICES

DATE OF REQUEST .-I-/- PROJECT NO. - - - - - XO NO. \_\_\_\_\_

1. FUND SOURCE (CIRCLE): P84 / DERA / DLA / \_\_\_\_\_ DIVISION: \_\_\_\_\_

2. INSTALLATION: \_\_\_\_\_

3. PROJECT OFFICER: \_\_\_\_\_ PHONE: \_\_\_\_\_

4. DESCRIPTION/PURPOSE (Screen, Regulatory/Health concern, etc.)  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

5. SAMPLE INFORMATION (SEE AEHA FORM 75-2,8-R, or 9-R)

6. SAMPLE CONTAINERS AND PRESERVATIVES (check one) [ ] NONE REQUIRED \_\_\_\_\_

[ ] Ship to: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

[ ] Picked up by Project Officer

7. DATE SAMPLES ARRIVED: -J-/- RESULTS NEEDED BY: -I-/-

8. REQUESTOR REMARKS AND SPECIAL REQUIREMENTS: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

9. LABORATORY REMARKS: ACCEPT [ ] REJECT [ ] and WHY? \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

10. DISTRIBUTION: C, LCSD [ ] C, RICO [ ] C, MAB [ ] C, NMAB [ ] C, OECD [ ]  
C, CAB [ ] C, PAB [ ] C, SAB [ ]

\_\_\_\_\_  
Program Manager

Date of Request — This is the date the form is completed.

Project Number — Self explanatory.

XO Number — Self explanatory.

1. **FUND SOURCE** — Circle the appropriate funding source. If not included in the list, write the source in the provided line.  
**DIVISION** -Write the division name of the requestor.
2. **INSTALLATION** —Write the name of the installation from which the samples are taken.
3. **PROJECT OFFICER** -The name of the person which will receive the sample data.  
**PHONE** — Self explanatory.
4. **DESCRIPTION/PURPOSE** — Project objective. Write a brief description on the reason for the survey or the requirement of the data. This will help the laboratory to understand how the samples should be analyzed.
5. **SAMPLE INFORMATION** — Self explanatory.
6. **PARAMETER/ANALYSES** — Indicate the chemical or group of chemicals that need analysis.
7. **MATRIX** —Write the sample matrix (soil, sludge, drinking water, natural water, etc.)
8. **-NUMBER SAMPLES** — Indicate the number of samples for that Parameter/Analyses and Matrix.
9. **REQUIREMENTS** — This space is available for special requirements (unusual detection limits requirements and/or required methods or pertinent comments).
10. **SAMPLE CONTAINERS AND PRESERVATIVES** -Self explanatory.  
**DATE REQUIRED** — This is the date the sampling supplies will be ready for pick-up or must be at the designated installation. Please give at least two weeks notice for all shipments.
11. **DATE SAMPLES ARRIVE** — This is the date the samples are anticipated to arrive in the laboratory.  
**RESULTS NEEDED BY** — This is the date the project officer needs the analysis data.
12. **REQUESTOR REMARKS AND SPECIAL REQUIREMENTS** -Write any remarks that may impact on sample analysis, i.e., chain-of-custody requirement.
13. **LABORATORY REMARKS** -This space is provided for comments that the laboratory personnel would like to express to the project officer.
14. **DISTRIBUTION** — Check appropriate laboratories that should receive this request.

*NOTE: For use of this form see USAEHA TG 155.*

## USAEHA TG No, 155

**(5) Collect and label samples.** Collect samples according to procedures discussed with laboratory. Follow all sample preservation instructions provided with sample containers. Label all samples with the following information:

- (a) Project Number.
- (b) Installation Name.
- (c) Sample Number.
- (d) Date Collected.
- (e) Preservative Added.
- (f) Required Analyses.

**(6) Transport/Submit samples to laboratories.** AEHA Form 75-2 (Listing of Specific Chemical Analyses), AEHA Form 8-R (Bulk Sample Data), and AEHA Form 9-R (Industrial Hygiene Air Sample Data) must be used as an inventory of samples as International Standards Organization (ISO) Guide 25 (section 11 .1 ) requirements. These forms and the samples should be delivered to AQAD for sample distribution/coordination. Figures F-3 through F-6 show examples of these forms. AEHA Form 8-R and AEHA Form 9-R will be reproduced locally. Copies of these forms for reproduction purposes is located at the back of this TG. **Alert AQAD and appropriate laboratory(s) if samples are to arrive on a weekend and need to be refrigerated and/or analyzed immediately!**

**NOTE: Check with the individual laboratories for potential additional form requirements**

### F-2, SPECIAL REQUIREMENTS

a. Both the project officer and the laboratory may have specific needs/requirements. Good communication will ensure that all such requirements are fully understood by both parties. Some types of "special requirements" are discussed below:

**(1) Unaccepted requests.** If a laboratory is unable to accept a request, the project officer should refer to the program manager for further direction/assistance.

**(2) Quick turn-around times.** If the turn-around time for laboratory analysis is not acceptable to the project officer, the project officer should refer to the program manager for direction/assistance.

**(3) Changes to request.** Changes to the original request should be discussed with laboratory personnel prior to sample collection/submittal.

**(4) 48-hour holding times.** Notify the laboratory by telephone 2 days in advance of arrival of samples requiring analyses within 48-hours.

**(5) Weekend arrivals.** Notify AQAD of any samples that will be arriving on the weekend and that require refrigeration or immediate analyses.

b. Most problems associated with a given project can be solved between the project officer and the laboratory. If not, refer to the program manager for assistance.

LISTING OF SPECIFIC CHEMICAL ANALYSES

Page  
of

A. REQUESTOR SAMPLE NO. →

B. LABORATORY NO. →

C. ANALYSIS ↓

**EXAMPLE**

REMARKS

AEHA Form 75-2, 1 Jun 81 (HSMB-LR)

Figure F-3. Example of a listing of Specific Chemical Analyses





| Calibration Information   |                     |          |                    |      |
|---|---------------------|----------|--------------------|------|
| Pump No.  | Calibration (L/min) |          | Rotometer Setting  | Date |
|   | Pre-Use             | Post-Use |                    |      |
|   |                     |          |                    |      |
|   |                     |          |                    |      |
|   |                     |          |                    |      |
|   |                     |          | Name of Calibrator |      |
| Operation   |                     |          |                    |      |
| Source of Contaminant:  |                     |          |                    |      |
|   |                     |          |                    |      |
| Operation Employee(s) Perform:  |                     |          |                    |      |
|   |                     |          |                    |      |
| Ventilation: <input type="checkbox"/> Local Exhaust <input type="checkbox"/> General Area <input type="checkbox"/> None |                     |          |                    |      |
| Personal Protective Equipment <i>(check if worn)</i>  |                     |          |                    |      |
| <input type="checkbox"/> Respiratory Protective Equipment    Type: _____  |                     |          |                    |      |
| <input type="checkbox"/> Protective Clothing    Type: _____   |                     |          |                    |      |
| <input type="checkbox"/> Gloves    Type: _____  |                     |          |                    |      |
| <input type="checkbox"/> Goggles/Face Shield  |                     |          |                    |      |
| <input type="checkbox"/> Ear Protection   |                     |          |                    |      |
| <input type="checkbox"/> Other: _____   |                     |          |                    |      |
| Field Notes/Additional Comments   |                     |          |                    |      |
|   |                     |          |                    |      |

EXAMPLE

Figure F-5. Example of an Industrial Hygiene Air Sample Data Form (Back)

**APPENDIX G****SOIL/SLUDGE SAMPLE PREPARATION FOR EXPLOSIVES AND/OR METALS ANALYSES**

---

**G-1. PURPOSE**

This appendix provides a step-by-step procedure for soil/sludge sample preparation for--

- a. High performance liquid chromatography (HPLC) analytical procedures for explosive analyses.
- b. Acid digestion of soil/sludge samples for the determination of metallic analytes.

**G-2. APPLICABILITY**

This appendix will be used by the USAEHA project officers and technicians who prepare soil/sludge samples for explosive and/or metals analyses.

**G-3. SAMPLE PREPARATION****a. Introduction.**

(1) The soil, sludge, or sediment samples are--

(a) Collected in I-L, wide-mouth glass jars with Teflon-lined caps.

(b) Shipped from the sample site to the USAEHA laboratory for analysis. Before the samples can be analyzed, the samples must meet certain moisture content and particle size requirements.

(2) Two of the common explosives used by the Army, namely tetryl and 2,4,6-trinitrotoluene (TNT), are degradable under sunlight.

(a) Samples for analysis of these compounds should be protected from prolonged light exposure during sample shipping and preparation.

(b) Note that tetryl has seldom been detected in soil and water samples collected and analyzed by USAEHA and has, therefore, been excluded from the routine suite of explosives requested for analyses.

(c) The compound trinitrobenzene (TNB) is now routinely requested as part of the explosives analyses.

(3) The extraction of the explosive compounds from soil/sludge samples--

(a) Occurs most easily when **the Samples are not too dry or hardened and are disaggregated** into small particle sites.

(b) Is most efficient when samples hold **2- to 3-percent** moisture and can pass through a 40-mesh sieve.

(4) The following explosives possess varying degrees of toxicity when inhaled, ingested, or absorbed through the skin. Therefore, direct skin contact, inhalation, or ingestion of these samples or sample dust should be avoided.

(a) Cyclotetramethylene tetranitramine (HMX).

(b) Cyclotrimethylenetrinitramine (RDX).

(c) TNB.

(d) 2,4,6-TNT.

(e) 2,4-dinitrotoluene (DNT) and 2,6-DNT.

**b. Sample drying area.**

(1) Soil, sludge, or sediment samples usually contain more than 2- to 3-percent moisture. A sample drying area is required to remove the excess moisture content.

(2) The drying area will--

(a) Consist of an enclosed area with adequate sample holding shelves and a dehumidifier. A hygrometer can be used to monitor the humidity if the dehumidifier has no (percentage) humidity setting.

(b) Be kept dark or shaded (except during sample handling and final preparation period).

(3) A sample preparation counter with a ventilation hood will be provided either in or nearby the drying area.

**c. Equipment required.**

(1) Scale.

(2) Sample spoon or spatula.

(3) Porcelain mortar and rubber-tipped pestle set.

(4) 40-mesh sieve.

(5) Cleaning brush.

(6) Funnel.

**d. Sample preparation procedures.**

**CAUTION: Wear vinyl gloves and a combination organic vapor/dust respirator, and work only under the ventilation hood area during sample preparation and equipment cleaning.**

(1) Scoop approximately 50 to 100 g of sample (depending on sample type and/or texture) from the sample jar and spread into a thin layer (approximately  $\frac{1}{8}$ -inch to  $\frac{1}{2}$ -inch thickness) on a clean sheet of paper.

(2) Place the soil samples on the drying shelves and turn on the dehumidifier or use a hygrometer to monitor the humidity in the drying area. If a dehumidifier is used, it should be set at approximately 40-percent humidity range.

(3) Keep the drying area dark or shaded during the sample drying period.

(4) Check the sample dryness every day. Soil samples normally take a few days to a week to dry; clay or oily sludges may take longer. Experience will tell if the sample is dried to 2- to 3-percent range, or use a moisture content test to confirm it.

(5) If the dried sample is not fine enough to pass through a 40-mesh sieve--

(a) Remove any stones or debris.

(b) Place the dried sample in a mortar under the ventilation hood.

(c) Disaggregate the sample with a rubber-tipped pestle to a finer form.

(6) Sieve the dried sample through a 40-mesh sieve.

**NOTE: For metals analyses alone, a 20- or 30-mesh sieve would be sufficiently fine.**

(7) Transfer a minimum of 20 g, preferably 30 to 40 g, of the sample into a glass bottle (such as a 2-oz Flint square bottle for dense samples or a 4-oz Flint square bottle for light samples, or equivalent).

(8) Cap with a Teflon or aluminum foil lid.

**NOTE: For metals analyses, the sample bottle must be capped with Teflon; an aluminum foil lid would be unacceptable.**

(9) Label the sample exactly how the original container was labeled in the field (see paragraph 3-3b).

(10) Clean the mortar and pestle thoroughly with a cleaning pad, tissue, or brush after each use. If necessary, wash and dry the mortar and pestle.

(11) Thoroughly brush clean the sieve after each use. This is done by turning the sieve upside down and brushing the bottom surface.

(12) Deliver the dried and sieved samples with the MFR, analytical requirements, and AEHA Form 75-2 to AQAD. Copies of the MFR and AEHA Form 75-2 should be furnished for the--

(a) Chromatography Analysis Branch, OECD.

(b) Metals Analysis Branch, RICD.

**NOTE: The sample holding time is >30 days.**

(13) After the dried samples are prepared, the remaining portion of the samples shall be kept until the sample analyses are completed. All samples shall be disposed of according to the analytical results.

**APPENDIX H**  
**SOIL SAMPLE PREPARATION**

---

**H-1.** Soil sample should be collected in a wide-mouth glass jar with a Teflon-lined cap and should be refrigerated to prevent biological degradation.

a. Sample volumes depend on the number of parameters requested and the texture and soil type. Normally a quart jar is suitable. Different parameters for the soil samples can be combined into the same container if they are of the same sieve size.

b. When VOCs are involved, the jar should be filled completely, leaving **as** little head space as possible.

**H-2.** Samples requiring inorganic analyses should be air dried except where samples are expected to contain highly volatile components which might be lost through the drying process (TOX, cyanide, and phenol).

a. The soils laboratory is equipped with a special area with low humidity for such preparation.

b. The final moisture content should be between 1 and 3 percent.

**H-3.** After drying, the large particles should be broken up with mortar and rubber tipped pestle.

a. Samples should be--

(1 ) Sieved through a 10-mesh sieve to remove rocks and debris.

(2) Well mixed to be as homogeneous as possible.

(3) Handled in a well ventilated hood. Gloves should be worn during handling.

b. Certain analyses are dependent on soil structure and these should be performed on the sample sieved to 10-mesh as described.

c. Other **analyses** depend on thorough digestion or complete extraction of a representative sample.

(1) In this case, samples should be ground farther with a mechanical grinder and passed through a 100-mesh sieve.

(2) If not thoroughly ground before sieving, the portion analyzed will not be representative of the actual sample.

d. Identify each container with the appropriate sieve size.

H-4. All apparatus must be thoroughly cleansed between samples.

- a. Wipe the mortar and pestle clean or, if necessary, wash and dry between use.
- b. Clean the sieve and mechanical grinder with a brush making sure to brush both sides of the screen.

H-5. Table H-I contains a summary chart showing which sieve sizes are suitable for which analyses.

H-6. Results will be reported on a dry weight basis unless otherwise specified.

H-7. Information concerning the soil structure can be useful to the laboratory in selecting a procedure particularly with regards to cation exchange capacity (CEC).

- a. Certain procedures are more suitable for acid soils and soils containing high carbonates or clays.
- b. Include, in your advanced memorandum, information on the suspected soil pH and type (high clay, vermiculite, or calcium carbonate (CaCO<sub>3</sub>) content).

Table H-1  
Summary Chart (Air Dry and Sieve)

| Analysis <sup>(a)</sup>                                      | Sieve Size                        | Minimum <sup>(b)</sup><br>Volume<br>(grams) |
|--|-----------------------------------|---|
| Alkalinity/Acidity (T-ALK)                                   | 10                                | 60  |
| Ammonia (NH <sub>3</sub> )                                   | 100                               | 30  |
| Base Saturation  | 10                                | 60  |
| Carbon, Total Organic (TOC)                                  | volatile, discuss with laboratory |   |
| COD  | volatile, discuss with laboratory |   |
| Cation Exchange Capacity (CEC)                               | 10                                | 60  |
| Chloride (Cl)  | 100                               | 60  |
| Conductivity (COND)  | 10                                | 60  |
| Cyanide (CN)   | volatile, discuss with laboratory |   |
| Exchangeable Acids   | 10                                | 60  |
| Exchangeable Cations (Bases)                                 | 10                                | 60  |
| Fluoride   | 100                               | 60  |
| Grease and Oil   | volatile, discuss with laboratory |   |
| Moisture <sup>(c)</sup>                                      | 10 & 100                          | 60  |
| Nitrate/nitrite Nitrogen (NO <sub>2</sub> /NO <sub>3</sub> ) | 100                               | 60  |
| Nitrogen, Total (TN)   | 100                               | 30  |
| Organic Matter   | volatile, discuss with laboratory |   |
| pH   | 10                                | 60  |
| Phenol   | volatile, discuss with laboratory |   |
| Phosphate, Total (TPO <sub>4</sub> )                         | 100                               | 30  |
| Plant Available K  | 10                                | 60  |
| Plant Available P  | 10                                | 60  |
| Plant Available N  | 10                                | 60  |
| Sulfate (SO <sub>4</sub> )                                   | 100                               | 60  |
| TOX  | volatile, discuss with laboratory |   |
| TKN  | 100                               | 60  |

<sup>(a)</sup> Those containing VOCs are typically best delivered to the laboratory untreated (not dried or sieved).

<sup>(b)</sup> The amount of sample listed includes enough volume to perform duplicate analyses and spikes of 10 percent of the samples. Contact laboratory for guidance if volume reduction is needed.

<sup>(c)</sup> Moisture must be performed on all sieve sizes used for sample preparations.

APPENDIX I

**POLYCHLORINATED BIPHENYLS (PCBS) AND BULK ANALYSIS  
SAMPLING PROCEDURES  
Transformers, Electrical Equipment, and Drums**

---

**I-1. GENERAL REQUIREMENTS**

a. Careless sampling procedures or contaminated sampling equipment can result in samples not truly representative of the overall material. Such samples may lead to erroneous conclusions and/or loss of time, effort, and expense involved in securing, transporting, and testing the sample.

b. This TG should be used as a guideline when obtaining samples of electrical insulating liquids from transformers or other enclosed containers (Appendix A, reference 52).

**I-2. EQUIPMENT, SAMPLING PROCEDURES, SAMPLE MANAGEMENT**

The following procedures are basic instructions that are to be followed with the sampling equipment (containers and pipettes) obtained through the AQAD of USAEHA. If possible, equipment should be obtained directly from USAEHA. In emergency situations, the installation/facility may use their own equipment (glass pipettes, tubing, and containers). The AQAD should be contacted at DSN 584-3269 or (410) 671-3269 for information on acceptable equipment.

**a. Equipment.**

(1) The sampling kit provided through USAEHA contains precleaned, ready-to-sample containers, sampling pipettes, and Teflon-lined flexible tubing. If other equipment is being used, its size, type (material), and cleanliness must be approved by AQAD.

(2) The installation/facility must supply all personnel conducting sampling with proper PPE. Disposable polyethylene gloves, boots, and Tyveks suits are recommended. Contact the installation safety office for appropriate PPE.

• • **IMPORTANT** • •

**AVOID SPILLS! Even small amounts of liquid may require specific clean-up procedures that result in the generation of regulated materials/wastes.**

• ☒☒☒☒☒☒☒☒☒☒

**b. Procedure.**

(1) Begin by putting on the appropriate PPE.

(2) Label sampling container(s) with the following information:

(a) Date.

(b) Installation/facility name.

(c) Type/description/specific location of equipment (to include transformer serial number).

(3) Evaluate the equipment to be sampled. If the provided equipment does not seem adequate for the necessary sampling, consult USAEHA. Otherwise, proceed with the appropriate sampling method as described below:

**c. Method.** The type of equipment being sampled will determine the sampling approach to be taken.

(1) **Equipment currently in-use** (transformers, capacitors, etc.) must first be de-energized before sampling! The liquid sample may be taken right from the surface with the pipette or through the vacuum/pressure release valve or other accessible opening with the flexible tubing.

**(a) Pipette method.**

1. Using gloves, submerge approximately two-thirds of pipette into liquid while **decompressing** rubber ball.

2. Release pressure on rubber ball and allow pipette to fill!

3. Remove and immediately place 5 mL of liquid into sampling container. Return excess liquid to equipment being sampled.

**(b) Tube method.**

1. Insert tubing through opening; ensure that tube reaches the container bottom.

2. Place thumb over end of tubing and carefully withdraw.

3. Using other hand, guide end of tube into sample container.

4. Slowly release liquid into sampling container filling about three-quarters. Return excess liquid to equipment being sampled.

**(21 Stored drums or equipment** contain liquids that may have “settled-out:” therefore, samples must be drawn from the lower depths of the liquid. This is best achieved through using a **COLIWASA** or drum thief. Follow the “Tube Method” described above.

**c. Preservation.** All samples should be stored in a cool place (4 °C) and protected from sunlight. Samples should not be stored for more than 24 hours before shipping for analysis.

**d. Packaging/Shipping.** Proper packaging is essential for this process to work.

(1) All samples should be placed in a Styrofoam packer. (Use the packer that the containers were originally sent in.)

(2) Along with a cold pack/freezer pack (such as Blue Ice -- **NOT** dry ice, as it cannot be shipped by air), wrap the Styrofoam packer with sufficient paper, etc. to protect during shipment. Place this package in a box or cooler and Federal Express overnight mail to USAEHA laboratories. (A box is sufficient for small quantities of samples; a cooler should be used when large numbers of samples are being sent.)

**f. Waste disposal.** Waste generated during sampling will include PPE, pipettes, and/or tubing. The PPE should be disposed of as general waste. Pipettes and tubing must be containerized and **labelled** as possible PCB items, pending analysis, with the sampling date on label. These items may be stored for 30 days until proper identification and disposal procedures are followed.

**g. Verification.** Within 48 hours, check with the laboratory at USAEHA to assure receipt of samples.

GLOSSARY

SECTION I - ABBREVIATIONS

|                         |  |
|-------------------------|--|
| AC                      | alternating current  |
| APED                    | Air Pollution Engineering Division   |
| <b>AQAD</b>             | Analytical Quality Assurance Division  |
| ASTM                    | American Society for Testing and Materials                                   |
|                         |  |
| BOD                     | biochemical oxygen demand  |
|                         |  |
| CAB                     | Chromatographic Analysis Branch  |
| <b>CaCO<sub>3</sub></b> | calcium carbonate  |
| CEC                     | cation exchange capacity   |
| CERCLA                  | Comprehensive Environmental Response, Compensation and Liability Act of 1980 |
| CFR                     | Code of Federal Regulations  |
| Cl                      | chloride   |
| <b>CLP</b>              | contract laboratory protocol   |
| cm                      | centimeter   |
| CN                      | cyanide  |
| COD                     | chemical oxygen demand   |
| <b>COLIWASA</b>         | composite liquid waste sampler   |
| COND                    | conductivity   |
| <b>CONUS</b>            | continental United States  |
| Cr <sup>+6</sup>        | hexavalent chromium  |
|                         |  |
| DA                      | Department of Army   |
| DC                      | direct current   |
| DEHE                    | Directorate of Environmental Health Engineering                              |
| DIH                     | Directorate of Industrial Hygiene  |
| DLS                     | Directorate of Laboratory Services   |
| DNT                     | dinitrotoluene   |
| DO                      | dissolved oxygen   |
| DOT                     | Department of Transportation   |
| DQO                     | data quality objectives  |
| DRES                    | Directorate of Radiation and Entomological Sciences                          |
| DSN                     | defense switched network   |
|                         |  |
| EP                      | Extraction Procedure   |
| EPA                     | U.S. Environmental Protection Agency   |
| ERT                     | emergency response team  |
|                         |  |
| FC                      | fecal coliform   |
| FS                      | fecal strep  |

|   |   |
|---|---|
| <b>g</b>  | gram  |
| <b>gal</b>                                      | gallon  |
| <b>GC-MS</b>                                    | gas chromatograph-mass spectrometry               |
| <b>gpd</b>                                      | gallons per day                                   |
| <b>gpm</b>                                      | gallons per minute                                |
| <b>HCl</b>                                      | hydrochloric acid                                 |
| <b>HM</b>                                       | hazardous material                                |
| <b>HMX</b>                                      | cyclotetramethylene tetranitramine                |
| <b>HNO<sub>3</sub></b>                          | nitric acid                                       |
| <b>HPLC</b>                                     | high performance liquid chromatograph             |
| <b>HW</b>                                       | hazardous waste                                   |
| <b>H<sub>2</sub>SO<sub>4</sub></b>              | sulfuric acid                                     |
| <b>ID</b>                                       | inside diameter                                   |
| <b>ISO</b>                                      | International Standards Organization              |
| <b>kg</b>                                       | kilogram  |
| <b>K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub></b> | potassium <b>dichromate</b>                       |
| <b>L</b>  | liter   |
| <b>LC</b>                                       | liquid chromatograph                              |
| <b>MAB</b>                                      | Metals Analysis Branch                            |
| <b>MCL</b>                                      | maximum contaminant level                         |
| <b>MEDDAC</b>                                   | medical department activity                       |
| <b>MFR</b>                                      | memorandum for record                             |
| <b>mg</b>                                       | milligram   |
| <b>MGD</b>                                      | million gallons per day                           |
| <b>mg/L</b>                                     | milligrams per liter                              |
| <b>mL</b>                                       | milliliter  |
| <b>mm</b>                                       | millimeter  |
| <b>N</b>  | normality   |
| <b>NaOH</b>                                     | sodium hydroxide                                  |
| <b>n.o.s.</b>                                   | not otherwise specified                           |
| <b>NH<sub>3</sub>-N</b>                         | Ammonia-nitrogen                                  |
| <b>NJDEP</b>                                    | New Jersey Department of Environmental Protection |
| <b>NMAB</b>                                     | Nonmetals Analysis Branch                         |
| <b>NO<sub>2</sub>-N</b>                         | nitrite-nitrogen                                  |
| <b>NO<sub>3</sub>-N</b>                         | nitrate-nitrogen                                  |
| <b>NO<sub>2</sub>NO<sub>3</sub></b>             | nitrite/nitrate nitrogen                          |
| <b>NPDES</b>                                    | National Pollutant Discharge Elimination System   |
| <b>NPDWR</b>                                    | National Primary Drinking Water Regulation        |
| <b>NSDWR</b>                                    | National Secondary Drinking Water Regulation      |

|                         |  |
|-------------------------|--|
| OB                      | open burning                                   |
| OCONUS                  | outside continental United States              |
| OD                      | open detonation                                |
| OECD                    | Organic Environmental Chemistry Division       |
| ORM                     | other regulated material                       |
| OSHA                    | Occupational Safety and Health Administration  |
| <b>oz</b>               | ounce  |
| <br>                    |  |
| PAB                     | Pesticides Analysis Branch                     |
| PCB                     | polychlorinated biphenyl                       |
| PL                      | Public Law                                     |
| <b>POC</b>              | point of contact                               |
| POX                     | purgeable organic halides                      |
| <b>PO<sub>4</sub>/P</b> | total phosphate                                |
| <b>ppb</b>              | parts per billion                              |
| PPE                     | personal protective equipment                  |
| <b>ppm</b>              | parts per million                              |
| <b>pt</b>               | pint   |
| PVC                     | polyvinyl chloride                             |
| PVNTMED                 | preventive medicine                            |
| <br>                    |  |
| QA                      | quality assurance                              |
| QC                      | quality control                                |
| <b>qt</b>               | quart  |
| <br>                    |  |
| RAB                     | Radiological Analysis Branch                   |
| RCRA                    | Resource Conservation and Recovery Act of 1976 |
| RDX                     | cyclotrimethylenetrinitramine                  |
| <b>RICD</b>             | Radiological and Inorganic Chemistry Division  |
| <br>                    |  |
| SAB                     | Special Analysis Branch                        |
| SCBA                    | self-contained breathing apparatus             |
| <b>STARLOG</b>          | sample tracking and reporting                  |
| <br>                    |  |
| T-ALK                   | total alkalinity                               |
| TCLP                    | Toxicity Characteristic Leaching Procedure     |
| TDS                     | total dissolved solids                         |
| TG                      | technical guide                                |
| THM                     | trihalomethane                                 |
| TKN                     | total Kjeldahl nitrogen                        |
| TN                      | total nitrogen                                 |
| TNB                     | trinitrobenzene                                |
| TNT                     | trinitrotoluene                                |
| TOC                     | total organic carbon                           |
| TOM                     | total organic matter                           |
| TON                     | total organic nitrogen                         |
| TOX                     | total organic halide                           |

|        |  |
|--------|--|
| TS     | total solids                           |
| TSCA   | Toxic Substances Control Act           |
| TSS    | total suspended solids                 |
| TURB   | turbidity                              |
| TVDS   | total volatile dissolved solids        |
| TVS    | total volatile solids                  |
| TVSS   | total volatile suspended solids        |
| UN     | United Nations                         |
| UPS    | United Parcel Service                  |
| USAEHA | U.S. Army Environmental Hygiene Agency |
| USCS   | Unified Soil Classification System     |
| USDA   | U.S. Department of Agriculture         |
| u x o  | unexploded ordinance                   |
| VOC    | volatile organic compound              |
| WDED   | Waste Disposal Engineering Division    |
| WQED   | Water Quality Engineering Division     |
| °C     | degrees centigrade (Celsius)           |
| μg     | microgram                              |
| μg/L   | micrograms per liter                   |

**SECTION II - TERMS**

|                   |  |
|-------------------|--|
| aerobic           | Requiring or pertaining to oxygen.   |
| aliquot           | A portion of a sample that is representative of the entire sample.   |
| anaerobic         | Requiring or pertaining to the absence of oxygen.  |
| background sample | A sample collected from an area similar to the one being studied, but in a site thought to be free of pollutants of concern. |
| bias              | The difference between the mean measurement and the true value.  |
| chain-of-custody  | Documentation of the possession and handling of a sample from the time it's collected to the final disposition.              |
| composite sample  | A nondiscreet sample representing more than one time, place, or condition.   |
| detection limit   | The lowest concentration at which a given analytical procedure can identify the presence of a specific parameter.            |

|                   |   |
|-------------------|---|
| duplicate samples | Samples collected simultaneously from the same source, under identical conditions, into separate containers.  |
| effluent          | Outflow or discharge.   |
| grab samples      | Samples which are discreet representations of conditions at a certain time and place.   |
| holding time      | The elapsed time between a sample's collection and the initiation of the analytical procedure.  |
| <b>influent</b>   | Inflow.   |
| preservation      | Techniques which retard physical and/or chemical changes in a sample after it has been collected.   |
| quality assurance | A monitoring program which ensures the production of quality data and identifies and quantifies all sources of error associated with each step of the sampling and analytical effort. |
| quality control   | The routine application of procedures which achieve the desired standard of quality.  |
| split sample      | A sample which has been portioned into two or more containers from a single sample container.   |

### SECTION III - TRADEMARKED NAMES

Use of trademarked names does not imply endorsement by the U.S. Army but is intended only to assist in identification of a specific product.

|                |   |
|----------------|---|
| <b>ISCO</b>    | Instrumental Specialties Co., Inc., Environmental Division, Lincoln, NE |
| <b>LAMINAR</b> | Troemner Inc., Philadelphia, PA   |
| Neoprene       | E.I. DuPont de Nemours & Co., Inc., Wilmington, DE                      |
| Ponar          | Wildlife Supply Company, Saginaw, MI                                    |
| Teflon         | E.I. DuPont de Nemours & Co., Inc., Wilmington, DE                      |
| Tyvek          | E.I. DuPont de Nemours & Co., Inc., Wilmington, DE                      |
| Whirlpak       | NASCO International, Inc., Fort Atkinson, WI                            |

**BULK SAMPLE DATA**

*For use of this form see USAEHA TG-141; the proponent is HSHB-DO.*

|   |  |
|---|--|
| Return Address <i>(complete address including Zip Code)</i> | Point of Contact <i>(name/AUTOVON)</i> |
|---|--|

|                      |                |   |  |  |  |  |  |  |
|----------------------|----------------|---|--|--|--|--|--|--|
| Sampled Installation | Project Number | ARLOC<br><table border="1" style="width:100%; height: 20px;"> <tr> <td style="width:15%;"></td> <td style="width:15%;"></td> <td style="width:15%;"></td> <td style="width:15%;"></td> <td style="width:15%;"></td> <td style="width:15%;"></td> </tr> </table> |  |  |  |  |  |  |
|                      |                |   |  |  |  |  |  |  |

|                      |                |              |
|----------------------|----------------|--------------|
| Samples Collected By | Date Collected | Date Shipped |
|----------------------|----------------|--------------|

|                          |                              |
|--------------------------|------------------------------|
| Description of Operation | Location <i>(BEDG, ALBA)</i> |
|--------------------------|------------------------------|

Associated Complaints *(be specific)*

Associated Air Samples *(see instructions for TD numbers)*  
 Yes     No

**Label Information**

|            |     |   |
|------------|-----|---|
| Trade Name | ASN | Manufacturer  |
| Address    |     | MSDS Attached<br><input type="checkbox"/> Yes <input type="checkbox"/> No |

Analysis Desired

| Lab Use<br>O n l y | Sample<br>No. | Constituents | Results | Remarks |
|--------------------|---------------|--------------|---------|---------|
|                    |               |              |         |         |
|                    |               |              |         |         |
|                    |               |              |         |         |
|                    |               |              |         |         |
|                    |               |              |         |         |
|                    |               |              |         |         |
|                    |               |              |         |         |
|                    |               |              |         |         |

Comment; to Lab:

**Lab Use Only**

|                           |                               |               |               |
|---------------------------|-------------------------------|---------------|---------------|
| Analyst <i>(initials)</i> | Reviewed By <i>(initials)</i> | Date Received | Date Reported |
| Procedures Performed      | Comments:                     |               |               |





**INDUSTRIAL HYGIENE AIR SAMPLE DATA**

*For use of this form see USAEHA TG 141; the proponent is HSEB-LO.*

|   |              |  |   |  |
|---|--------------|--|---|--|
| Return Address <i>(complete address including Zip Code)</i> |              |  | Point of Contact <i>(name/AUTOVON)</i>  |  |
| Samples Collected By  |              |  | Associated Bulk Samples<br><input type="checkbox"/> Yes <input type="checkbox"/> No<br>Bulk Sample No(s): |  |
| Date Collected  | Gate Shipped |  |   |  |

|                |                      |  |
|----------------|----------------------|--|
| Project Number | Sampled Installation | ARLOC<br><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |
|----------------|----------------------|--|

|                            |  |
|----------------------------|--|
| Location <i>(BDG/AREA)</i> | Description of Operation <i>(details on reverse)</i> |
|----------------------------|--|

|  |                                  |                      |
|--|----------------------------------|----------------------|
| <input type="checkbox"/> Persons Exposed | <input type="checkbox"/> Hrs/Day | Method of Collection |
|--|----------------------------------|----------------------|

Associated Complaints *(be specific) (state NONE if applicable)*

Analysis Desired

| Sampling Data    |  |  |  |  |  |                       |
|------------------|--|--|--|--|--|-----------------------|
| Sample No.       |  |  |  |  |  |                       |
| Pump No.         |  |  |  |  |  |                       |
| Time On          |  |  |  |  |  | B<br>L<br>A<br>N<br>K |
| Time Off         |  |  |  |  |  |                       |
| Total Time (min) |  |  |  |  |  |                       |
| Flow Rate (LPM)  |  |  |  |  |  |                       |
| Volume (Liters)  |  |  |  |  |  |                       |
| GA/SZ            |  |  |  |  |  |                       |
| Employee Name/ID |  |  |  |  |  |                       |
| Laboratory No.   |  |  |  |  |  |                       |

| Results |  |  |  |  |  |  |
|---------|--|--|--|--|--|--|
|         |  |  |  |  |  |  |
|         |  |  |  |  |  |  |
|         |  |  |  |  |  |  |
|         |  |  |  |  |  |  |
|         |  |  |  |  |  |  |
|         |  |  |  |  |  |  |

Comments to Lab:

| Lab Use Only              |                               |               |                 |
|---------------------------|-------------------------------|---------------|-----------------|
| Analyst <i>(initials)</i> | Reviewed By <i>(initials)</i> | Date Received | Date Dispatched |

**Calibration Information**

| Pump No. | Calibration (L/min) |          | Rotometer Setting | Date |
|----------|---------------------|----------|-------------------|------|
|          | Pre-Use             | Post-Use |                   |      |
|          |                     |          |                   |      |
|          |                     |          |                   |      |
|          |                     |          |                   |      |
|          |                     |          |                   |      |
|          |                     |          |                   |      |

 Name of Calibrator \_\_\_\_\_

**Operation**

Source of Contaminant: \_\_\_\_\_

Operation Employee(s) Perform: \_\_\_\_\_

Ventilation:       Local Exhaust       General Area       None

**Personal Protective Equipment (check if worn)**

- Respiratory Protective Equipment    Type: \_\_\_\_\_
- Protective Clothing    Type: \_\_\_\_\_
- Gloves    Type: \_\_\_\_\_
- Goggles/Face Shield
- Ear Protection
- Other: \_\_\_\_\_

**Field Notes/Additional Comments**

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SAMPLE ALTERATION FORM  
*The Proponent of this form is the Waste Disposal Engineering Division*

*OTE: For use of this form see USAEHATG155.*

Project Name and Number: \_\_\_\_\_

Materials to be Sampled: \_\_\_\_\_

Measurement Parameter: \_\_\_\_\_

**STANDARD PROCEDURE FOR FIELD COLLECTION AND LABORATORY ANALYSIS (cite references):**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

4. \_\_\_\_\_

**REASON FOR CHANGE IN FIELD PROCEDURE**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

4. \_\_\_\_\_

**VARIATION FOR FIELD PROCEDURE**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

4. \_\_\_\_\_

**SPECIAL EQUIPMENT MATERIALS. OR PERSONNEL REQUIRED**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

4. \_\_\_\_\_

Initiator's Name \_\_\_\_\_ Date: \_\_\_\_\_

Project Approval \_\_\_\_\_ Date: \_\_\_\_\_

Laboratory Approval \_\_\_\_\_ Date: \_\_\_\_\_

Sample Control Center \_\_\_\_\_ Date: \_\_\_\_\_

REQUEST-FOR LABORATORY SERVICES

DATE OF REQUEST . - / - / - PROJECT NO. \_\_\_\_\_ XO NO. \_\_\_\_\_

FUND SOURCE (CIRCLE): P84 / DERA / DLA / \_\_\_\_\_ DIVISION: \_\_\_\_\_

2. INSTALLATION: \_\_\_\_\_

3. PROJECT OFFICER: \_\_\_\_\_ PHONE: \_\_\_\_\_

4. DESCRIPTION/PURPOSE (Screen, Regulatory/Health concern, etc.)  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

5. SAMPLE INFORMATION (SEE AEHA FORM 75-2, 8-R, or 9-R)

6. SAMPLE CONTAINERS AND PRESERVATIVES (check one) DATE REQUIRED \_\_\_\_/\_\_\_\_/\_\_\_\_

Ship to \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Picked up by Project Officer \_\_\_\_\_

7. DATE SAMPLES ARRIVED: \_\_\_\_/\_\_\_\_/\_\_\_\_ - RESULTS NEEDED BY: \_\_\_\_/\_\_\_\_/\_\_\_\_

8. REQUESTOR REMARKS AND SPECIAL REQUIREMENTS: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

9. LABORATORY REMARKS: ACCEPT  REJECT  and WHY? \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

10. DISTRIBUTION: C, LCSD  C, RICD  C, MAB  C, NMAB  C, OECD   
C, CAB  C, PAB  C, SAB

\_\_\_\_\_  
Program Manager

Date of Request — This is the date the form is completed.

Project Number — Self explanatory.

XO Number-Self explanatory.

1. FUND SOURCE — Circle the appropriate funding source. If not included in the list, write the source in the provided line.  
DIVISION — Write the division name of the requestor.
2. INSTALLATION — Write the name of the installation from which the samples are taken.
3. PROJECT OFFICER -The name of the person which will receive the sample data.  
PHONE — Self explanatory.
4. DESCRIPTION/PURPOSE — Project objective. Write a brief description on the reason for the survey or the requirement of the data. This will help the laboratory to understand how the samples should be analyzed.
5. SAMPLE INFORMATION — Self explanatory.
6. PARAMETER/ANALYSES — Indicate the chemical or group of chemicals that need analysis.
7. MATRIX -Write the sample matrix (soil, sludge, drinking water, natural water, etc.)
8. NUMBER SAMPLES — Indicate the number of samples for that Parameter/Analyses and Matrix.
9. REQUIREMENTS\* - This space is available for special requirements (unusual detection limits requirement and/or required methods or pertinent comments).
10. SAMPLE CONTAINERS AND PRESERVATIVES — Self explanatory..  
DATE REQUIRED — This is the date the sampling supplies will be ready for pick-up or must be at the designated installation. Please give at least two weeks notice for all shipments.
11. DATE SAMPLES ARRIVE — This is the date the samples are anticipated to arrive in the laboratory.  
RESULTS NEEDED BY — This is the date the project officer needs the analysis data.
12. REQUESTOR REMARKS AND SPECIAL REQUIREMENTS -Write any remarks that may impact on sample analysis, i.e., chain-of-custody requirement.
13. LABORATORY REMARKS — This space is provided for comments that the laboratory personnel would like to express to the project officer.
14. DISTRIBUTION — Check appropriate laboratories that should receive this request.

NOTE: For use of this form see USAEHA TG 155.

**FIELD CORRECTIVE ACTION FORM**  
*The Proponent of this form is the Waste Disposal Engineering Division*

*NOTE For use of this form see USAEHA TG 165.*

Project Name and Number: \_\_\_\_\_

Sample Data Involved: \_\_\_\_\_

Measurement Parameter(s): \_\_\_\_\_

Acceptable Data Range: \_\_\_\_\_

**I. PROBLEM AREAS REQUIRING CORRECTIVE ACTION**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

**II. MEASURES REQUIRED TO CORRECT PROBLEMS**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

**III. MEANS OF DETECTING PROBLEMS**

1. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

Initiator's Name \_\_\_\_\_ Date: \_\_\_\_\_

Project Approval: \_\_\_\_\_ Date: \_\_\_\_\_

Laboratory Approval: \_\_\_\_\_ Date: \_\_\_\_\_

QA Officer/Reviewer: \_\_\_\_\_ Date: \_\_\_\_\_

Sample Control Center: \_\_\_\_\_ Date: \_\_\_\_\_

DIRECTORATE OF LABORATORY SCIENCES CORRECTIVE ACTION FORM

NOTE: For use of this form see USAEHA TG 155.

1. DATE(S): \_\_\_\_\_ REPORT NO. \_\_\_\_\_

2. FILED BY: \_\_\_\_\_

3. PROBLEM(S): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

4. WHAT DATA WAS IMPACTED? (N/A - IF NONE) \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

5. MAJOR AREAS/SITUATIONS INVESTIGATED: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

6. CONCLUSIONS OF INVESTIGATION: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

7. CORRECTIVE ACTIONS TAKEN - DATE: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

BRANCH APPROVAL: \_\_\_\_\_ DATE: \_\_\_\_/\_\_\_\_/\_\_\_\_

DIVISION APPROVAL: \_\_\_\_\_ DATE: \_\_\_\_/\_\_\_\_/\_\_\_\_

DIRECTOR APPROVAL: \_\_\_\_\_ DATE: \_\_\_\_/\_\_\_\_/\_\_\_\_

FOLLOW-UP INSPECTION FINDINGS; \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

INSPECTOR: \_\_\_\_\_ DATE: \_\_\_\_/\_\_\_\_/\_\_\_\_

FEBRUARY 1993



USAEHA **TG** No. **155**