# CHAPTER 7

# SAMPLE COLLECTION PROCEDURES

**ESSENTIAL ELEMENTS OF A SAMPLING PLAN**

- Goals of the Sampling Plan
- Description of the Facility Generating Sludge
- Data Quality Objectives
- Selecting and Describing Sampling Points
- **Sample Collection Procedures**
- Sample Handling Procedures
- Evaluation of Completeness
- Record-Keeping and Reporting Procedures

*After establishing data quality objectives and selecting appropriate sampling points, the next step in developing a sampling plan is to establish and describe the sample collection procedure. To ensure consistency, all elements involved in sample collection must be included. Prior to sampling, all sampling equipment and procedures, as well as methods of cleaning and preparing sampling equipment and containers, should be determined. Safety equipment and precautions should be described. This attention to detail will help minimize potential errors.*

## Equipment

Sludge and biosolids can have a wide range of physical characteristics. Solids content can range from 1 percent to over 90 percent. As a result, consistency can vary from liquid, to mud-like, to a dry pelletized solid. The equipment needed to sample a particular material must be appropriate for the physical properties of your sludge.

*Sludge consistency can vary from a low percent solids (L) to a high percent solids (R).*

### Equipment types

Sampling equipment is generally divided into samplers used for liquids or solids. Listed below are some commonly used and commercially available sampling devices for collecting sludge samples.

***The equipment needed to sample a particular material must be appropriate for the physical properties of your sludge.***

- **Common devices used for sampling liquid or flowable material**

**Graduated Cylinders or Pitchers:** Vessels made of glass, plastic, or stainless steel can be used to sample liquid or semi-solid sludge collected from taps. The vessel can be attached to a pole or other device to extend its reach or to sample from open channels.


*Graduated Cylinder*

**Composite Liquid Sludge Sampler:** A composite liquid sludge sampler or “coliwasa” can be used to collect flowable sludge from a lagoon, tank, or other sludge containment areas. A “core” or depth profile can be collected with this device. The coliwasa is typically a 5-foot long (longer versions are commercially available) tube fabricated from metal, glass, or plastic. The bottom is fitted with a mechanism that can be opened and closed to collect a sample when the coliwasa is lowered into liquid sludge. To use the device, the bottom is opened and slowly lowered into the sludge. Once the intended depth is reached, the bottom of the coliwasa is closed and the sample is collected. The sample collected is a composite of the sludge profile at that point. A “sludge judge” is a similar device that is typically used to measure the depth of the sludge blanket in a lagoon or tank. However, after measuring the depth of the blanket, a sample of sludge can be collected for analysis.

*A sludge judge can be used to collect sludge samples.*

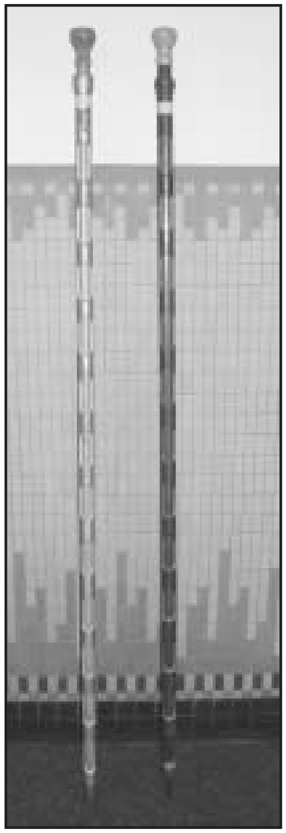
- **Common devices used for sampling solid or semi-solid material**

**Thief Sampler**: This is most appropriately used for drier and more granular sludge products such as compost or pellets. It is a useful tool for collecting a core sample through the depth of a mass of material. The device is composed of two slotted tubes, one of which fits inside the other, and typically made of metal. Rotating the inner tube closes the sampler. A similar sampling device known as a “Missouri D probe” can also be used to collect dry or granular sludge.

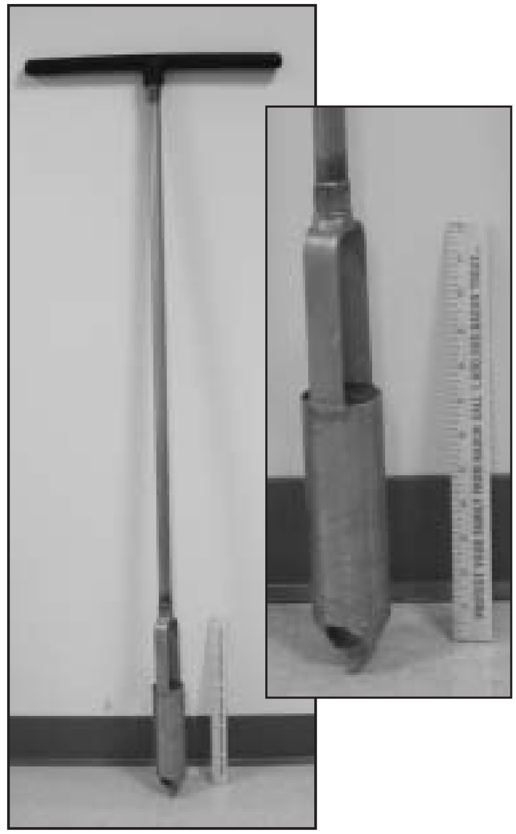
To use this type of sampling device, the thief is pushed into the material to be sampled with the slots opened and oriented upward. Typically, the thief has a pointed or tapered end to facilitate penetration. Once the thief has reached the desired depth, the inner tube is rotated to close the slots and collect the material.

**Trier:** This device is most useful for sampling dewatered sludge that has manure or mud-like consistency. Like the thief, a trier is ideal for collecting core samples and is

*Missouri D Probe*



*Trier*



*Soil Auger, note drill-like cutters*

used in a similar manner. The trier is comprised of a single metal tube, generally stainless steel or brass, which has been cut in half along its length. The end of the tube is sharpened to allow the trier to be more easily pushed into the material being sampled. Commercially available probes for soil sampling have a similar construction and can be used for the same purpose.

**Auger:** A soil auger can be used to collect sludge samples that have hardened or been compressed. The auger is particularly useful when a trier or thief cannot be successfully pushed into the material being sampled. Augers typically consist of a metal handle and extension attached to spiral drill-like metal blades. The auger is used to drill into the sludge to collect a sample. The material can be collected over the entire depth of the drilled hole to form a composite sample, or the material can be collected from a specific depth in the sludge for a grab sample.

*Other commonly-used sampling tools*

**Shovels and Scoops**: These tools are useful for sampling granulated, powdered, or other loose sludge. They are particularly convenient to use when dewatered sludge is being conveyed within the POTW.

**Other Equipment:** The tools described above are employed for the actual collection of sludge samples. However, a variety of other items are needed for sampling biosolids. Table 7-1 provides a list of typical sludge sampling equipment.

**Table 7-1. TYPICAL SLUDGE SAMPLING EQUIPMENT**

| |
|---|
| **Protective Gear:** |
| Disposable gloves (such as nitrile or latex)<br>Tyvek sleeves<br>Face shield or other appropriate eye protection |
| **Sample handling:** |
| Bucket (accumulate and mix grab samples)<br>Tongue depressors (transferring sludge)<br>Small stainless steel hand trowel (transferring or mixing sludge) |
| **Cleaning equipment:** |
| Disposable towels<br>Soap, such as a low-phosphate laboratory detergent<br>Scrub brush<br>Rinse water<br>Deionized water<br>Tarp or plastic sheets<br>Foil or other protective wrap |
| **Sample ID, marking, and labeling:** |
| Labels for sample containers<br>Custody seals<br>Pens, pencils, markers<br>Chain-of-custody form(s)<br>Field notebook or sample log |
| **Transporting and Preservation:** |
| Sample containers<br>Cooler<br>Ice |

### Equipment Selection

All equipment that is used to collect and prepare sludge samples must be prepared so that it does not contaminate or react with the material being sampled. Contamination can arise if equipment is improperly cleaned or is made of materials that are released into the sample. (Galvanized or chrome-plated implements must be avoided.) Relatively inert materials such as Teflon, glass, or stainless steel are typically used for sampling equipment or containers. However, Teflon and stainless steel are expensive, and glass is heavy and fragile. In certain situations, plastic, non-stainless steel, or aluminum sampling equipment can be used in place of the preferred materials. However, care should be taken when using these substitute materials because sample contamination could result when used inappropriately. For example, if a sludge sample is collected for metals analysis, a plastic sampling device or container is acceptable; the plastic, however, can contaminate a sample being tested for semi-volatile compounds. Sampling equipment needs to be chosen based on the analysis being performed as well as the consistency of the material being sampled.

***Sampling equipment needs to be chosen based on the analysis being performed as well as the consistency of the material being sampled.***

### Equipment Preparation and Cleaning

Before using sampling equipment for the first time and after every use, it must be thoroughly cleaned. Cleaning procedures may differ slightly, depending on the type of sampling equipment and the analysis to be performed. For ease of cleaning, it is best to clean equipment as soon as possible after use, or at least to perform a preliminary rinse to remove gross contamination. Sludge can be very sticky and hard to remove, especially after it has dried and hardened.

Below is a generalized cleaning procedure that can be used to prepare sampling equipment between sampling events:

1. Rinse equipment with warm tap water to remove the majority of solids.
2. Using a brush and standard low-phosphate lab detergent, scrub the equipment to remove all residues.
3. After scrubbing, triple rinse the equipment with tap water.
4. For the final rinse, triple rinse with deionized water.
5. When sampling for microbial parameters, sterilize the sampling equipment by exposing to high pressure steam of at least 121° C for at least 15 minutes.

Depending on your data quality objectives, it may be necessary to further decontaminate the sampling equipment with various cleaning solutions, depending on the particular analysis to be performed. If metals analyses employing stringent QA/QC objectives are to be performed, rinsing with a dilute acid such as 10 percent nitric acid prior to the final deionized water rinse is recommended.

If organic constituents are the target, an organic solvent such as methanol may be an appropriate decontaminant. Depending on the compounds of interest and the level of contamination in samples previously collected, it may be necessary to employ additional organic solvents to ensure thorough decontamination. Regardless of the organic solvent(s) used, it is best to conduct the final organic solvent rinse with a water-miscible solvent such as methanol.

Any decontaminant solution should be compatible with the material it is being used to clean to avoid damaging the sampling equipment. When using these additional decontamination rinses, take special safety precautions and properly dispose of the used rinsate, which may be considered a hazardous waste and must be disposed of accordingly.

This procedure can and should be modified to meet the needs of each specific sampling program and its established data quality objectives. Upon completion of the cleaning procedure, air-dry the equipment and then wrap it in an inert material such as aluminum foil to protect it until the next use.

If the decontamination process is performed in the field, any rinsing done with acids or organic solvents should be contained. The used organic rinse solutions should be collected separately, containerized, and transported to the lab for proper disposal.

To clean sampling equipment that does not come in contact with sampled material, a detergent wash followed by triple rinsing with tap water and then deionized water is sufficient. ASTM D5088 (*Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*) provides detailed guidance on equipment cleaning and decontamination procedures.

As mentioned in Chapter 5, to ensure that cleaning procedures and techniques are adequate, equipment blanks should be collected and analyzed for analytes of interest. To collect an equipment blank, soak sampling equipment in deionized water overnight. The water should then be removed from contact with the sampling device, collected, and analyzed for potential contaminants.

***As mentioned in Chapter 5, to ensure that cleaning procedures and techniques are adequate, equipment blanks should be collected and analyzed for analytes of interest.***

## Sample Containers

Like sampling equipment, sample containers should not add contaminants to or react with the sampled material they hold. Sample integrity should not be compromised by the addition or removal of analytes because of a container.

### Compatibility and Cleaning

Generally, sample containers are made of glass or plastic because these materials are relatively inert and easily cleaned. Glass is a good choice for sampling containers. The drawback in using glass containers is that they can be heavy and are easily broken. Plastic containers have the advantage of lighter weight and greater durability; however, they are generally not suitable for samples subject to analysis for organic compounds because of the potential for phthalate contamination or adsorption of the target analyte to the sample container. Glass is the best choice for organic constituents, but covers or caps should be lined with Teflon.

*Glass wide-mouth jars are commonly used for sludge samples.*

The cleaning procedure for sampling equipment is also applicable to sampling containers. The cleaning protocol should be tailored to meet the data quality objectives for the subject analysis. A quality control assessment of the cleaning procedure should be performed. This can be accomplished by storing deionized water in previously cleaned container and analyzing the water from the container during a subsequent round of testing.

Ideally, certified pre-cleaned, laboratory-grade containers should be provided by the laboratory doing the sample analysis, thus eliminating the need for container cleaning by sample collection staff.

Although not strictly a compatibility or cleanliness issue, it is most convenient to use wide-mouth bottles, particularly for samples of sticky material. Wide-mouth jars are also easier to clean. The size of the sample container must be large enough to hold an adequate amount of material for the test being performed. In fact, it is preferable if the container is slightly oversized so it holds enough material to also perform replicate or laboratory QC sample analyses.

***Although not strictly a compatibility or cleanliness issue, it is most convenient to use wide-mouth bottles, particularly for samples of sticky material.***

The types of containers needed for particular analytical methods and the appropriate cleaning and preparation procedures are typically specified in approved EPA analytical methods. If analytical services are conducted by an outside environmental laboratory, sample bottles are frequently provided by the lab. Sampling staff should verify that the bottles and their preparation are appropriate for the particular analytical method to be employed and that they meet the data quality objectives of the sampling plan.

Table 7-2 provides general recommendations for sample containers for a variety of common sludge analyses. Appendix D shows the specific analyses required by states in the Northeast. It also includes requirements for sample containers.

**Table 7-2. GENERAL RECOMMENDATIONS FOR SAMPLE CONTAINERS**

| Test | Recommended Container/Size |
|---|---|
| pH | 250 mL glass or plastic |
| Solids | 250 mL glass or plastic |
| Mercury | 250 mL glass or plastic |
| Other 503 metals | 500 mL glass or plastic |
| Total phosphorus | 250 mL glass or plastic |
| Nitrogen (nitrate, TKN, ammonia) | 250 mL glass or plastic |
| Potassium | 250 mL glass or plastic |
| Volatile Organic Compounds | 40 mL glass vial with Teflon cap liner |
| Semi-volatile Organic Compounds | 250 mL glass with Teflon cap liner |
| Pesticides/PCB | 250 mL glass with Teflon cap liner |
| Dioxin | 250 mL glass with Teflon cap liner |
| Microbiological test, FC, Salmonella, etc. | 250 mL sterile plastic or glass |

## Sampling Procedures

This phase in the development of a sampling plan involves preparation of standard operating procedures for implementation in the field. Significant planning has already established sampling objectives, data quality objectives, sampling points, and other procedures regarding the collection of a sample. Your description of sampling procedures should outline the actual procedures that will be employed before, during, and after the collection of a sample. In order to adequately describe the sampling procedures and promote consistency, the sampling plan should include an equipment checklist and a written standard operating procedure (as described below) that can be carried into the field.

***Your description of sampling procedures should outline the actual procedures that will be employed before, during, and after the collection of a sample.***

**Equipment Checklist** – a convenient and reliable means for ensuring that sampling staff remember to bring everything needed for a successful sampling effort. Having the necessary equipment clean and in good working order prior to arriving at the sampling site saves time and frustration and is more likely to result in consistent sampling procedures, which in turn results in more accurate and reliable analytical data. Some categories of equipment and specific items that might be included in an equipment checklist are listed in Table 7-1. Appendix H contains an example of an equipment checklist.

**Standard Operating Procedure (SOP)** – a standardized set of sampling procedures that promotes consistent sampling and sample handling for each event and enhances data comparability. Appendix H provides an example SOP for sampling sludge from a POTW. An initial SOP should be a first attempt to describe sampling procedures and is an important part of any sampling plan. However, the SOP should not be a static document and should be reviewed and revised as procedures are improved or changed.

# Standard Operating Procedures

It is recommended that an SOP address the following issues:

***Preparation for Sampling***

Steps need to be taken prior to beginning the sampling process. When preparing for a sampling event, the following key considerations should be addressed:

- Notify the lab performing the analyses and schedule the sampling event.
- Assemble and/or clean sampling implements.
- Assemble and/or clean sample containers.
- Assemble and prepare any sample handling equipment (coolers, labels, notebooks, custody forms, markers).

Checklists are very useful in this portion of the SOP.

***Sampling Procedures***

Previous segments of the sampling plan discuss sampling points, sample type (grab versus composite) and other key criteria. The SOP is the means by which these criteria are conveyed to sampling staff or personnel who may have occasional sampling responsibilities. The SOP should inform those collecting samples about the particulars of the sampling process, such as what, when, where, and how sampling activities should be conducted. Important details determined in other parts of the sampling plan that are relevant to the actual process of collecting a sample should be included in the SOP. The SOP should also include safety procedures necessary to protect sampling staff from potential risk associated with the material to be sampled and the procedures for sample collection. Instructions relative to field QA/QC samples should also be provided in the SOP.

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***Sampling Handling, After Collection***

This portion of the SOP should specify how samples are processed and handled after collection. Details relevant to the integrity, validity and documentation of the sample after collection should be covered. These procedures should already have been determined, but the SOP describes how they will be implemented. Key information to convey in this section includes:

- How the sample will be labeled and sealed.
- What information should be recorded in sampling field notes.
- How the sample will be preserved and transported.
- How chain-of-custody is maintained and documented.

See Chapters 8 and 9 for more information on post-collection sample handling.

## Safety

An important component of any sampling plan, process, or event is the procedures developed and implemented to protect sampling personnel from the potential hazards of sludge sampling. Some of these hazards are typical of those faced by all individuals working at wastewater treatment facilities. The equipment, machinery, and working environment at any wastewater treatment plant present inherent risks that must be addressed to protect the health and safety of the workers. Safety concerns for all POTW operators, regardless of their specific job function, include:

***An important component of any sampling plan, process, or event is the procedures developed and implemented to protect sampling personnel from the potential hazards of sludge sampling.***

- Burns, electrocution, or injury from machinery or chemicals used in wastewater treatment processes.
- Explosion or asphyxiation from gases in confined spaces.
- Illness from exposure to pathogens in wastewater.

All POTWs have safety plans or protocols that apply to the entire facility. These plans identify the sources of risk and impose procedures to minimize or eliminate the hazard. Such safety protocols should be cited in the sampling plan to provide procedures for reducing the risk of injury. These safety protocols can also serve as a reference and basis for safety procedures specified in the sampling plan. Safety procedures should address the following issues:

- Identification of potential hazards
- Identification of protective gear and its use
- Personal hygiene procedures and standard practice to reduce pathogen risk
- Consideration of the need for immunization against certain diseases
- Other specific practices necessary to avoid injury or illness

Wastewater treatment plant operators face a variety of potential injury and illness risks. Moisture can create slipping and falling hazards. Limbs can be caught in machinery. Heat and pressure associated with some treatment processes may cause burns. Many of these concerns are common to any industrial workplace. Individuals involved in the collection of sludge samples can be at risk for some of these common industrial accidents plus any pathogens that exist in wastewater and sludge. Personal protective equipment, such as gloves and face shields, are needed to reduce exposure to these pathogens. Immunization should be considered to prevent diseases such as tetanus and hepatitis. Also, training in appropriate personnel hygiene during and after sampling is important.

More information about safety precautions that should be taken when sampling and analyzing samples can be found in *Standard Methods for the Examination of Water and Wastewater* (APHA, 1992) in Sections 1060A and 1090C.

## Documentation

Documentation of sample collection efforts is important, especially when enforcement or legal action could be associated with a particular set of samples. Also, the interpretation of analytical results can be facilitated by an understanding of the conditions under which samples were collected. Most frequently, however, sample documentation in the form of field notes and sample labels verifies that samples were collected according to established SOPs. An example of a field data sheet is contained in Appendix I.

### Field Records

Field notes or logbooks that document a sampling event should include the following:

- Sample identification
- Sample location (sampling point)
- Type of sample (composite or grab, number of grab samples and, for continuous processes, the interval between grab samples; for composite samples, the number of grab samples collected and their relative weighting)
- Sampling equipment and a brief description of sampling procedure
- Date and time of collection
- Weather conditions
- Analyses required
- Notes on unusual conditions or deviations from established protocols

## Sample Labeling

After a sample is collected, it is placed into a container or containers that are compatible with the intended analyses. Sample collection sheets or chain-of-custody sheets are used to identify samples for analysis in the lab. However, sample containers themselves must be labeled to correspond to the information recorded on the custody sheet. Also, it is advisable that the sample containers be labeled in a manner that clearly identifies the sample without referring to the custody sheet. The following information should be included on container labels:

- Sample identification
- Date and time of collection
- Sample type (grab or composite)
- Sample location
- Person collecting sample
- Preservative
- Required test(s)

## CHAPTER 7 REFERENCES:

*Standard Methods for the Examination of Water and Wastewater.* American Public Health Association. 1992.

*An Addendum to the POTW Sludge Sampling and Analysis Guidance Document.* Gaines, Cristina and Safavi, Behzad. US EPA, Office of Wastewater Enforcement and Compliance. May 1992.

*POTW Sludge Sampling and Analysis Guidance Document.* Permits Division, Office of Water, Washington, DC 20460. August 1989.

*Process Design Manual: Land Application of Sewage Sludge and Domestic Septage,* EPA/625/R-95/001. US EPA, Office of Research and Development. September 1995.

*Standard Practices for Decontamination of Field Equipment Used at Waste Sites,* D 5088-02. ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. 2002.

*Sludge Survey Methods for Anaerobic Lagoons.* North Carolina Cooperative Extension Service. North Carolina State University. 2003.

*AAPFCO Inspection Manual, 6th ed., Chapter 11, Sampling Methods,* p.83.