NEIWPCC

The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans

Prepared by the
New England Interstate Water Pollution Control Commission

September 2006

This page is intentionally blank.

The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans

September 2006

Prepared by the

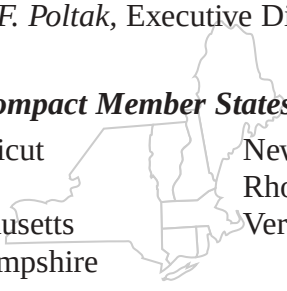
NEW ENGLAND INTERSTATE WATER
POLLUTION CONTROL COMMISSION
116 John Street ■ Lowell, MA 01852-1124

Tel: (978) 323-7929 ■ Fax: (978) 323-7919
mail@neiwpcc.org ■ www.neiwpcc.org

Ronald F. Poltak, Executive Director

Compact Member States

Connecticut
Maine
Massachusetts
New Hampshire
New York
Rhode Island
Vermont



For additional copies, contact NEIWPCC at the address above.

This document is also available for download at www.neiwpcc.org.

♻ *Printed on recycled paper*

This page is intentionally blank.

ACKNOWLEDGEMENTS

This manual was developed by the New England Interstate Water Pollution Control Commission. NEIWPCC is a not-for-profit interstate agency, established by an Act of Congress in 1947, which serves its member states (Connecticut, Maine, Massachusetts, New Hampshire, New York, Rhode Island, and Vermont) by providing coordination, public education, research, training, and leadership in water management and protection. This manual was made possible by a grant from the U.S. Environmental Protection Agency (EPA). Thelma Murphy served as the EPA project officer for the grant and this project. The contents do not necessarily reflect the views and policies of EPA or NEIWPCC's member states, nor does the mention of trade names or commercial products or processes constitute endorsement or recommendation for use.

This manual was compiled and written under the direction of an advisory committee consisting of representatives of NEIWPCC member state environmental agencies and EPA. Special thanks to Michael Rainey for his invaluable assistance in developing this guide.

Advisory Committee:

Gary Johnson, Connecticut DEP
Michael Clark, Maine DEP
Mark Casella, Massachusetts DEP
Anthony Pilawski, New Jersey DEP
Michael Rainey, New Hampshire DES
Ly Lim and Sally Rowland, New York State DEC
Alex Pinto, Rhode Island DEM
Cathy Jamieson and Ernie Kelley, Vermont DEC
Thelma Murphy, U.S. EPA - Region 1

NEIWPCC would like to thank the following people who contributed their time in reviewing this manual.

Robert B. Brobst, P.E., U.S. EPA - Region 8
Mark C. Meckes, U.S. EPA, Office of Research and Development
Dr. James E. Smith, Jr., U.S. EPA, Office of Research and Development
Rick Stevens, U.S. EPA, Office of Science and Technology
Jorja DuFresne, Minnesota Pollution Control Agency
Greg Kester, Wisconsin DNR
Robert Rubin, NCSU-BAE and McKim & Creed
Charles Button, Massachusetts Water Resources Authority
Michael Delaney, Massachusetts Water Resources Authority

NEIWPCC Project Officer
Michael Jennings

Design and Production
Ricki Pappo, Enosis – The Environmental Outreach Group

Editorial Support
Ellen Frye, Enosis – The Environmental Outreach Group
Stephen Hochbrunn, NEIWPCC

Cover Photo: *Todd Weber of the Lowell, Massachusetts Regional Wastewater Utility collects dewatered sludge.*

This page is intentionally blank.

HOW TO USE THIS DOCUMENT

The development of a sampling plan should be the first step in the implementation of a well-run sampling program. This guidance document is intended to help wastewater treatment plant operators develop a comprehensive sludge (or biosolids) sampling plan. Unlike other sludge sampling guidance documents that provide general recommendations, this guide was developed to be as specific as possible and provide easily adaptable worksheets and appendices that can be utilized to form a suitable sludge sampling plan.

Wastewater treatment plant operators generally have limited time and/or experience to devote to developing sampling plans—hence the need for this document. Although reading this guidance document from cover to cover prior to starting the sampling plan development process is highly encouraged, it is probably an unrealistic expectation. Therefore, to convey useful information effectively and efficiently, this document has been designed to be used as a quick reference to assist wastewater treatment plant operators in completing the *Sampling Plan Worksheet* found in Appendix A. Once completed, the worksheet will contain the appropriate information consistent with a comprehensive sampling plan.

The *Sampling Plan Worksheet* makes reference to specific chapters and appendices that can be found in this guide and used to complete the specific sections of the worksheet. Many of the guide's appendices can be photocopied and attached to complete applicable sections of the *Sampling Plan Worksheet.*

Follow these steps to complete a sampling plan for your facility:

1. Photocopy or obtain an electronic copy of the *Sampling Plan Worksheet* found in Appendix A.

2. Read Chapter 1 – *Introduction*, and Chapter 2 – *Elements of a Sampling Plan*, in the main document to gain a general understanding of what a sampling plan is and the elements it should contain.

3. Complete the *Sampling Plan Worksheet* using the main document as a reference.
   - When in doubt, refer to the specific chapters and appendices cited in the individual sections of the worksheet to adequately complete the information requested.
   - Where needed, photocopy applicable appendices or tables to help complete relevant sections of the worksheet.

This page is intentionally blank.

CONTENTS

ACKNOWLEDGEMENTS i
HOW TO USE THIS DOUCMENT iii
CONTENTS v

CHAPTER 1: INTRODUCTION

Introduction. 1-1
Why Collect Samples? 1-2
Role of the Sampling Plan 1-2
Chapter 1 References. 1-2

CHAPTER 2: ELEMENTS OF A SAMPLING PLAN

Goals of the Sampling Plan (Chapter 3) 2-1
Description of the Facility Generating Sludge (Chapter 4) 2-2
Data Quality Objectives (Chapter 5) 2-2
Selection and Description of Sampling Points (Chapter 6) 2-3
Sample Collection Procedures (Chapter 7) 2-3
Sample Handling Procedures (Chapter 8) 2-3
Evaluation of Completeness (Chapter 9) 2-3
Record-Keeping and Reporting Procedures (Chapter 10) 2-3
Chapter 2 References. 2-4

CHAPTER 3: GOALS OF A SAMPLING PLAN

Goals of the Sampling Plan 3-1
Chapter 3 References. 3-2

CHAPTER 4: DESCRIPTION OF THE FACILITY GENERATING SLUDGE

Wastewater Treatment Process 4-1
Sludge Treatment and Handling Process 4-3
Chapter 4 References. 4-5

CHAPTER 5: DATA QUALITY OBJECTIVES

Desired or Required Analytes to be Tested 5-2
Analytical Methods and Protocols 5-2
Quality Assurance/Quality Standards and Procedures 5-3
Sample Type, Frequency, and Size 5-7
Costs of Sampling and Analysis 5-11
Laboratory Qualifications 5-11
Chapter 5 References. 5-12

CHAPTER 6: SELECTION AND DESCRIPTION OF SAMPLING POINTS

Representativeness 6-1
Type of Process—Batch or Continuous 6-2
Accessibility 6-2
Safety 6-3
Typical Sampling Points 6-3
Chapter 6 References. 6-4

CHAPTER 7: SAMPLE COLLECTION PROCEDURES

Equipment 7-1
Sample Containers 7-6
Sampling Procedures 7-8
Safety 7-10
Documentation 7-11
Sample Labeling 7-11
Chapter 7 References. 7-12

CHAPTER 8: SAMPLE HANDLING PROCEDURES

Sample Preservation 8-1
Sample Holding Times 8-1
Chain of Custody 8-4
Submittal of Samples 8-4
Chain-of-Custody Record Form 8-6
Transportation of Samples 8-6
At the Laboratory 8-8
Chapter 8 References. 8-8

CHAPTER 9: EVALUATION OF COMPLETENESS

Data Review 9-1
Review of Sampling Procedures 9-2
Overall Review of Your Sampling Program 9-3
Chapter 9 References. 9-4

CHAPTER 10: RECORD-KEEPING AND REPORTING PROCEDURES

General Considerations 10-1
Record-Keeping for Sludge Preparers 10-3
Reporting Requirements for Sludge Preparers 10-4
Other Record-Keeping or Reporting Requirements 10-4
Chapter 10 References. 10-5

APPENDICES

A: Sampling Plan Worksheet A-1
B: Example of a Completed Sampling Plan Worksheet B-1
C: Pathogen Reduction and Vector Attraction Reduction (PR/VAR) Methods and Options C-1
D: State and Federal Analytical Requirements D-1
E: Questions to Assist with Laboratory Selection E-1
F: Microbial Sampling Considerations F-1
G: Student's T-Test Calculation G-1
H: Example of Sludge Sampling Procedure H-1
I: Field Data Sheet I-1
J: Example Chain-of-Custody Form J-1
K: Glossary K-1
L: Regional Regulatory Contact Information L-1

FEEDBACK FORM

This page is intentionally blank.

CHAPTER 1
INTRODUCTION

Sewage sludge is the residue generated during the treatment of domestic sewage. This residue can be used as a soil conditioner or fertilizer component if properly treated and utilized in a manner that protects human health and the environment. Throughout the wastewater industry, the term "sewage sludge" has largely been replaced by the term "biosolids," which specifically refers to sewage sludge that has undergone treatment and meets federal and state standards for beneficial reuse. Throughout this document the terms "sewage sludge," "sludge," "biosolids," and the generic term "solids" are used interchangeably.

Throughout the wastewater industry, the term "sewage sludge" has largely been replaced by the term "biosolids," which specifically refers to sewage sludge that has undergone treatment and meets federal and state standards for beneficial reuse.

Biosolids have been successfully applied to agricultural land (e.g., pastures, cropland), disturbed areas (e.g., mined land, construction sites), plant nurseries, forests, recreational areas (e.g., parks, golf courses), cemeteries, highway and airport runway medians, and home lawns and gardens.

Operators of publicly owned treatment works (POTWs) where treated sewage sludge is processed for land application are required by state and federal regulations to analyze this material for specific chemical, physical, and biological parameters to ensure that it is in compliance with applicable standards. State biosolids regulators may also wish to collect biosolids samples from both treatment facilities and land application sites prior to spreading.

The purpose of this document is to provide guidance and standard operating procedures for the collection and analysis of municipal sewage sludge or biosolids. The information provided herein will be applicable to sampling both at POTWs and at land application sites where biosolids are stockpiled.

The Sampling Program

Most biosolids sampling programs provide a framework for obtaining samples that represent the chemical, physical, and biological characteristics of sludge material that is being land applied. A representative sample depends on a number of factors including the analyses required, the material sampled, the sampling location, and the objective of the sampling program.

Why Collect Samples?

Most POTW sampling programs are initiated to demonstrate compliance with state and/or federal regulations. As an operator, it is important that you understand exactly what a regulatory entity requires to demonstrate compliance, particularly at the state level. For example, some states regulate biosolids under their solid waste statutes, not their wastewater programs. Accordingly, while a solid waste regulation may require Toxicity Characteristic Leaching Procedure (TCLP) metals analysis, the federal Part 503 regulations call for total metals analysis. In addition, many state regulatory programs have varying target compounds, analytical protocols that are more stringent than federal protocols, and specific detection limits.

Another potential objective of a biosolids sampling effort is to assess the variability of biosolids relative to chemical, physical, and microbial quality. This information can be particularly important in terms of public acceptance and confidence in a beneficial use program. For example, the public needs to know that biosolids are relatively consistent in terms of potential toxic constituents (i.e., heavy metals, polychlorinated biphenyls (PCBs), or dioxins). End users, such as farmers, want to know that they are receiving a consistent product that will perform predictably. Regulators need to be certain that a given quantity of material meets regulatory standards. Elected or appointed officials may also want information confirming safety and efficacy of local government. In addition, POTW operators may want to routinely monitor sludge for information or trends that will assist them in making operational or maintenance decisions within the treatment process.

Role of the Sampling Plan

The development of a sampling plan is the first step in the implementation of an effective sampling program.

The development of a sampling plan is the first step in the implementation of an effective sampling program. While it is understandable that analytical studies, with their sophisticated instrumentation and high cost, are often perceived as the dominant element in a biosolids characterization program, analytical data generated by a scientifically defective sampling plan have limited utility, particularly in regulatory proceedings. Inappropriately collected samples yield incorrect results. Therefore, it is important to ensure that samples are always collected and handled properly.

CHAPTER 1 REFERENCES

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

Sampling/Analysis Work Plan Guidance. Maine Department of Environmental Protection, 17 State House Station, Augusta, ME. September 16, 2005.

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

CHAPTER 2
ELEMENTS OF A SAMPLING PLAN

A sampling plan is a blueprint for how a sampling event or program will be executed. It should provide all the detail needed to ensure that representative samples are collected, handled, analyzed, and reported in a manner that meets the needs and objectives of the sampler (e.g., POTW, regulator, third-party auditors). Implementing a clearly defined and consistently employed sampling protocol reduces the chance that the sampling process will be a source of error.

Implementing a clearly defined and consistently employed sampling protocol reduces the chance that the sampling process will be a source of error.

Inappropriate or inconsistent sampling techniques or procedures have an impact on the accuracy and precision of analytical results. Accuracy is a measure of how closely testing results reflect the actual chemical, physical, and biological properties of the biosolids sampled. Precision is a measure of the variability of data associated with a specific sludge quality parameter. Inaccurate or imprecise analytical data may falsely indicate compliance or violation of regulatory requirements and result in flawed decisions.

Under a National Pollutant Discharge Elimination System (NPDES) permit or a state permit, the owner/operators of a treatment works are ultimately responsible for the quality of the data they report to the permitting authority. Given the potential unintended consequences of poor data quality, the value of using a sampling plan to optimize data integrity is evident.

The United States Environmental Protection Agency (EPA) and many states have developed guidance documents that outline the essential elements of a sampling plan (referred to in some documents as a “sampling and analysis plan” or an “analysis plan”). This document discusses the essential elements of a sampling plan, derived from numerous guidance documents. A sampling plan worksheet is included in Appendix A and an example of a completed sampling plan is included in Appendix B.

Goals of the Sampling Plan (Chapter 3)

The goals or objectives of a sampling plan should describe what you hope to accomplish by implementing a sampling program. For example: Will the data be used for process control? Are the data intended to demonstrate compliance with state and federal regulations? Is the operator evaluating sludge quality in order to decide on an appropriate sludge management option? In each of these examples, sludge variability and contaminant sources would need to be evaluated or identified.

Description of the Facility Generating Sludge (Chapter 4)

A facility description (including a flow diagram or schematic) should provide an overview of the configuration and operation of the facility that is generating the sludge to be tested. The physical, chemical, and biological properties of the solids produced by a facility are determined by several factors:

- Influent wastewater characteristics and treatment
- Sludge handling (i.e., wasting, mixing, holding, thickening), and dewatering processes
- Treatment methods used to achieve pathogen reduction (PR) and vector attraction reduction (VAR)

If land application is the final solids management option, this section should also include details on how the material is handled prior to land application. For instance, is it stored or stockpiled prior to land application, and if so, how long is it stockpiled, and where and under what conditions is the material stored?

Data Quality Objectives (Chapter 5)

Your data quality objectives should state the standards and specifications for the data you will generate. The primary goal is for the data to be as representative of the actual sludge quality as possible. Determining data quality objectives during the development of a sampling plan may take the most planning and research. It is essential that those preparing the sampling plan are knowledgeable about lab procedures and protocols. In this regard, it is imperative that you communicate with the laboratory or laboratories that will be conducting the analyses during the development and implementation of the sampling plan.

Determining data quality objectives during the development of a sampling plan may take the most planning and research.

The data quality objectives should include the following elements:

- Clear identification and discussion of issues such as detection limits, precision, accuracy, comparability, and completeness
- Analytical protocols required for each test (e.g., metals, fecal coliform, nutrients) that will be performed
- Data reduction, validation, and reporting methods
- Data quality objectives for field measurements or goals for field Quality Assurance/Quality Control (QA/QC)
- Type of sample (i.e., grab or composite) that will be collected at each sampling point
- Process for producing composite samples including how grab samples are weighted
- Sample size or volume of grabs and composite samples
- Frequency and timing of sample collection

Selection and Description of Sampling Points (Chapter 6)

Your sampling point(s) should be accurately and specifically described relative to location and utilization. Accessibility and safety must be considered in the selection of these points. The description should explain how the sampling points were chosen in order to produce a representative sample and how specific points will meet the goals of the sampling program.

Sample Collection Procedures (Chapter 7)

Step-by-step instructions on sample collection procedures must be developed and documented in writing. This written procedure is frequently referred to as a standard operating procedure or "SOP." SOPs should include detailed information on the following elements:

- Type of equipment used for sampling
- Methods for cleaning and decontaminating sampling equipment
- Sample collection process
- Sample identification and documentation

Sample Handling Procedures (Chapter 8)

This section focuses on sample handling after collection. Sample preservation and holding times, Chain-of-custody procedures, and sample transportation procedures (including shipping and storage) should be described in detail. These parameters must be determined and described for each type of sample and analysis.

Evaluation of Completeness (Chapter 9)

Evaluating data completeness refers to the process of determining whether the goals of the sampling program have been met. This process answers such questions as: Have data quality objectives been met? Were the samples properly preserved and handled? Were reporting requirements satisfied? This process also provides an opportunity to evaluate the sampling plan itself. Does the sampling plan or protocol need revision? Do the data quality objectives need to be adjusted?

Record-Keeping and Reporting Procedures (Chapter 10)

The last section of your sampling plan should describe what data and information will be retained, how it will be stored and retrieved, and for how long it will be retained. Reporting requirements should also be addressed. Is reporting required and by whom? Criteria such as reporting units, data validation procedures, and reporting format should be determined during the development of the sampling program.

The remainder of this document will describe each of these elements in greater detail. Wherever possible, a range of options will be provided, and the benefits or drawbacks of each option will be described.

CHAPTER 2 REFERENCES

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

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

CHAPTER 3
GOALS OF THE SAMPLING PLAN

ESSENTIAL ELEMENTS OF A SAMPLING PLAN

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

To begin development of your sampling plan, the goal or purpose of your facility's sampling program must be clearly identified and articulated. Most state or federal sludge-related guidance documents suggest that collecting a representative sample is the foremost goal of any sampling effort. Your sampling plan should seek to ensure that representative samples are collected and that the analytical data generated are a true reflection of the chemical, biological, or physical characteristics of the material being evaluated.

Your sampling plan should seek to ensure that representative samples are collected and that the analytical data generated are a true reflection of the chemical, biological, or physical characteristics of the material being evaluated.

Goals of the Sampling Plan

The goals and objectives of the sampling effort should describe what your facility hopes to accomplish by implementing the sampling program. For example: Will the data be used for process control? Is the data intended to demonstrate compliance with state and federal regulations? Is the operator evaluating the sludge quality in order to decide on the appropriate sludge management options? In some instances there may be multiple goals and objectives for the sampling program. Evaluating sewage sludge can include the following objectives:

- Determine if a sewage sludge is suitable for land application
- Assess ongoing compliance with state or federal regulations
- Evaluate variability of biosolids in terms of their chemical, physical, and biological characteristics
- Determine the most cost-effective sludge-management options.

This section of the sampling plan need not be long or complex. A single sentence reflecting the reason for implementing your sampling program may be sufficient.

For example:

"The goal of our sludge sampling program, as detailed in this sampling plan, is to demonstrate compliance with state and federal biosolids regulations."

This concise statement is more than adequate as a starting point for developing a sampling plan. In fact, POTWs typically institute sludge sampling programs to demonstrate compliance with state and federal biosolids regulations.

The goal(s) of your sampling program will lead to (or in some cases dictate) the elements needed within the sampling plan. If a demonstration of compliance with the federal Part 503 regulations for land application is the goal, then the sampling plan must contain all of the necessary elements to demonstrate compliance with federal regulations that require chemical contaminant sampling and analysis for nine metals (arsenic, cadmium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc). Samples may also need to be collected and analyzed to demonstrate that pathogen and vector attraction reduction requirements have been met. A sampling program designed to demonstrate compliance with state-specific land application regulations may require more elaborate sampling and chemical analysis and a more elaborate sampling plan. In summary, simple, clearly defined goals are necessary to develop a focused and coherent sampling plan.

CHAPTER 3 REFERENCES

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

Sampling/Analysis Work Plan Guidance. Maine Department of Environmental Protection, 17 State House Station, Augusta, ME. September 16, 2005.

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

CHAPTER 4
DESCRIPTION OF THE FACILITY GENERATING SLUDGE

ESSENTIAL ELEMENTS OF A SAMPLING PLAN

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

This section of your sampling plan should provide an overview of the configuration and operation of the facility generating the sludge to be tested. The physical, chemical, and biological characteristics of solids produced by POTWs are determined by the wastewater treatment (i.e., influent wastewater characteristics, type of wastewater treatment) and sludge treatment and handling processes.

Wastewater Treatment Process

A thorough and detailed description of your entire wastewater treatment process provides important information necessary to the development of your sampling plan. A complete description of the wastewater treatment process from the headworks through disinfection will provide information pertinent to the selection of sampling points, collection of a representative sample, and interpretation of analytical results. The description of your wastewater treatment process should include the following items:

A complete description of the wastewater treatment process from the headworks through disinfection will provide information pertinent to the selection of sampling points, collection of a representative sample, and interpretation of analytical results.

General Description: Identify the type of treatment at your facility and the permitted design capacity (in million gallons per day – mgd) of the facility. For example, is the facility an aerated lagoon, rotating biological contactor (RBC), or conventional activated sludge facility? A flow diagram or schematic is a useful way to provide a general description of the facility and its treatment processes. The flow diagram or schematic should also identify the sample collection sites.

Industrial Pretreatment: Although industrial pretreatment is not a unit process at a POTW, it is a function of wastewater treatment that can have a profound effect on sludge quality. Effective industrial pretreatment is one of the foremost reasons why land application of biosolids has

become a sustainable recycling practice. Since industrial pretreatment keeps many pollutants out of wastewater, effective pretreatment reduces the pollutant loading in sludge. Your facility description should include an overview of the industrial pretreatment program or sewer use ordinance including local limits, inspection schedules, and types and number of industries discharging to the collection system.

Screening/Comminution: Describe the location, sequence, and type of screens, racks, or grinders that your facility may employ in the treatment process.

Grit Removal: Describe the type of grit removal mechanism used at your facility and how the grit is disposed. For example, is the grit removed in an aerated grit chamber or by a horizontal-flow grit chamber?

Flow Equalization: Provide information on whether your wastewater treatment facility uses flow equalization to maintain a constant hydraulic or biochemical oxygen demand (BOD) loading rate. If flow equalization is used, this section should describe whether the equalization basins are in-line or off-line and what type of aeration or mixing is used in the basins (if any).

Other Pretreatment Operations: Indicate whether your facility employs other pretreatment operations prior to primary settling. These operations generally involve skimming, flocculation, or preaeration. Skimming tanks are designed to remove solids with a density less than water (i.e., floating material). Facilities that receive a high grease load might have a skimmer. Flocculation is used to increase settling in primary or secondary settling tanks or to condition certain industrial wastes. Numerous objectives are given for preaeration prior to primary settling. Generally, these objectives involve improved treatability of wastewater in subsequent processes. Regardless of the objectives, many of these pretreatment operations can have an impact on sludge quantities and generation rates.

Primary Sedimentation: It is important to note whether your facility has primary settling tanks and, if so, to describe their configuration and operation. Many smaller facilities, such as extended aeration plants and aerated lagoons, do not have primary settling. Consequently, such facilities will not have sludge from primary sedimentation, also known as primary sludge. The presence (or lack of) primary sludge will significantly affect the odor properties of resulting biosolids. Other facilities may have flotation or screening for removal of solids. Imhoff tanks and septic tanks are used by some smaller facilities. A significant consideration to address in your facility description is whether the digestion or degradation of solids or BOD is taking place during the sedimentation process or during the handling of primary sludge after settling.

Biological Treatment: Most modern wastewater treatment plants include at least secondary treatment, which involves the use of a biological reactor for the degradation of BOD and suspended solids. This section should include information that describes the process for biological treatment. For example, describe whether biological treatment is based on fixed-bed media or a complete-mix system. If it is a fixed media, state whether the reactor is a trickling filter or RBC. If the system is complete-mix, state whether it is an extended air system, oxidation ditch, or lagoon. If the system is a lagoon, state whether it is it aerated or facultative. For activated sludge facilities, describe how the reactor operates—plug flow, continuous flow, stepped aeration, or contact stabilization. All of these factors can have an impact on sludge quality and generation rates.

Secondary Sedimentation: Secondary sedimentation or settling is an integral part of the activated sludge process. Secondary clarifiers are designed to separate activated sludge from mixed liquor. Flocculating solids in the aeration basin promotes settling and consequently the removal of suspended solids and BOD. The settled biological solids can be recycled into the reactor or wasted and sent to the sludge treatment process. How and when solids are recycled and/or wasted can affect sludge quality and generation rates.

Advanced Treatment: Increasingly, POTWs are faced with upgrading their facilities to include advanced wastewater treatment in order to reduce nutrient loading to receiving waters. Generally, advanced treatment is designed to address lower effluent discharge permit limits for nitrogen or phosphorus. In addition, any chemicals added for nutrient removal (e.g., alum) should be described. Metals removal may also be a treatment goal. It is particularly important that you describe the advanced treatment processes used at your facility to increase the volume of solids generated.

Disinfection and Miscellaneous Treatment Processes: After final clarification, the wastewater treatment process generally culminates with a disinfection step prior to discharge to the receiving water. The method of disinfection does not significantly impact sludge quality or characteristics, but it should be noted to complete the facility description. The facility description should also discuss any processes that are not necessarily common to all plants. For example, odor controls may be extensive at some treatment works but nonexistent at others. Treatment plants may have very different needs in terms of the types of unit operations that are used to control odors. Additionally, the choice of odor control technology may vary among facilities. Odor treatment utilizing chemical oxidation of the wastewater can have an influence on sludge chemistry. Therefore it is critical that all treatment processes are described, at least briefly, so they can be evaluated for potential impacts on sludge quality or characteristics.

Sludge Treatment and Handling Process

While wastewater treatment operations certainly can have an effect on solids properties, the processes involving direct manipulation of wastewater sludge have the greatest potential to impact quality. A thorough description of the sludge treatment and handling process is a critical step in preparing your sludge sampling plan. The following considerations should be included in your description of sludge treatment and handling processes:

A thorough description of the sludge treatment and handling process is a critical step in preparing your sludge sampling plan.

Solids Sources: Depending on the type of facility, solids can be contributed from any of several unit operations within the POTW. The types of sludge and the proportion of each in the final mixture have a significant impact on sludge quality and odor properties. For example, primary sludge is highly putresible. Primary sludge or a sludge that has a high percentage of sludge from primary settling or clarification has a greater potential to cause malodor than a digested sludge. Also, sludge that results from advanced treatment for nutrient removal can have greater nitrogen or phosphorus content and an associated higher value as a fertilizer or soil conditioner. All solids sources and their relative proportions within the sludge treatment and handling process must be described in detail.

Preliminary Operations: In general, preliminary operations include pumping, blending, storage, and in some cases thickening of solids prior to dewatering or other processes. Also, as more facilities are required to have advanced treatment, sludge blending becomes important (relative to the production of a consistent material for dewatering and/or land application). In addition, some facilities find it necessary to employ sludge grinding or degritting prior to subsequent processes, based on the configuration of the facility or the characteristics of the wastewater. Most POTWs, with the exception of lagoon systems, have the ability to store sludge prior to dewatering operations or disposal. The conditions under which sludge is stored can impact both its dewatering capability and its chemical quality as a soil conditioner. For example, some facilities aerate stored sludge, while others employ gravity thickeners that double as storage tanks. In some cases, thickeners (e.g., flotation thickeners) are used prior to storage and dewatering. All preliminary sludge operations must be described in detail.

Pathogen Reduction/Vector Attraction Reduction (PR/VAR): Other than the character of the wastewater being treated, the treatment processes used for PR/VAR are probably the single biggest factor influencing sludge quality, and must be described in detail. The federal Part 503 regulations establish the requirements for PR/VAR prior to the use or disposal of sewage sludge. During the development and implementation of the Part 503 regulations, EPA published a variety of guidance documents describing the operation standards and tests necessary to achieve Class A or Class B pathogen-reduction status. Pathogen reduction and vector attraction reduction methods and options are listed in Appendix C.

The following documents discuss treatment processes that are acceptable for PR/VAR:

- "Standards for the Use or Disposal of Sewage Sludge; Final Rules," *Federal Register,* Friday, February 19, 1993.
- *Part 503 Implementation Guidance,* EPA 833-R-95-001, October 1995, US EPA, Office of Water.
- *Control of Pathogens and Vector Attraction in Sewage Sludge,* EPA/625/R-92/013, July 2003, US EPA, Office of Research and Development.
- *A Plain English Guide to the EPA Part 503 Biosolids Rule.* EPA/832/R-93/003, September 1994, US EPA, Office of Wastewater Management.

These documents describe acceptable treatment processes and specify the testing, documentation, and record-keeping necessary to demonstrate proper PR/VAR. Citing the pathogen reduction alternative and vector attraction reduction option (along with specific treatment objectives of these requirements) employed at your facility will adequately identify the sludge treatment process. Pathogen and vector attraction reduction alternatives and options are listed in Appendix C.

Dewatering: There are a variety of methods for reducing the water content of sludge. Dewatering is typically accomplished via filter presses, centrifugation, or drying beds or lagoons. Heat-drying dewaters sludge and can also be used to meet pathogen and vector attraction reduction requirements, if the appropriate operational standards are achieved. As would be expected, the method of dewatering has a strong impact on the physical properties of sludge. Also, there is increasing evidence that the chemicals used to condition sludge and improve dewatering can

have a significant influence on sludge chemistry and even odor potential. The dewatering methods (including chemical additions) must be described in detail.

Disposal or Recycling Option: The handling and storage of treated sludge after PR/VAR can have an impact on sludge quality and odor properties, particularly for undigested materials; therefore, the methods of disposal and/or recycling must be described in detail. For example, some heat-dried biosolids can become malodorous after being rewetted, and lime-stabilized biosolids can cause odor problems if stored long enough for the pH to drop (allowing microbial activity to resume). For compost curing, federal regulations require testing after prolonged storage. If land application is your final solids management option, this section should also include details on how sludge is handled prior to land application. For instance, if the material is stored or stockpiled prior to land application, you need to describe how long, where, and under what conditions the material is stored.

CHAPTER 4 REFERENCES

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

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge. US EPA, Office of Research and Development, EPA/625/R-92/013. Revised July 2003.

Examination of Mechanisms for Odor Compound Generation During Lime Stabilization, Kim et al. Water Environment Research, Vol. 75, No. 2, 121-125pp.

Guide to Field Storage of Biosolids. US EPA, Office of Wastewater Management, EPA/832-B-00-007, July 2000.

Wastewater Engineering; Treatment, Disposal, and Reuse. Metcalf & Eddy, Inc. Third Edition. 1991.

This page is intentionally blank.

CHAPTER 5
DATA QUALITY OBJECTIVES

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

Your data quality objectives (DQOs) represent the primary planning phase of your sampling plan development. In this section, your task is to state as clearly as possible what the data should look like and the minimum quality standards those data must meet. As stated previously, your overall goal or goals for the sampling plan have a strong influence on your quality standards and the need to document data quality. For example, if you are assessing compliance with regulations, you may need more clearly defined and definitively documented data than if you were using sampling data for process control. Once the quality standards are set, the methods for evaluating the quality of the data and determining if quality standards have been met should be specified.

The process of developing data quality objectives has the added benefit of serving as the basis for a request for proposal (RFP) in the procurement of laboratory services.

The process of setting DQOs and measuring to determine if those objectives have been met is frequently referred to as quality assurance/quality control (QA/QC). The QA/QC process is not just applicable to the analytical portion of a sampling program. Field measurement and sampling procedures must also conform to standards established prior to any actual sampling event. All sampling and analytical methods, procedures, and controls should serve to enhance the probability that representative samples are collected and that the analytical results accurately describe the quality of the sludge from which the samples originated. In addition, the process of developing data quality objectives has the added benefit of serving as the basis for a request for proposal (RFP) in the procurement of laboratory services.

The issues that need to be addressed in this section of the sampling plan are:

- *Desired or required analytes to be tested*
- *Analytical methods or protocols*
- *QA/QC standards and procedures*
- *Sample type, frequency, and size*
- *Cost of sampling and analysis*

Desired or Required Analytes to be Tested

If the goal of your sampling program is to demonstrate compliance, then state and/or federal regulations determine the list of analytes for which sampling must be performed. Be aware that your biosolids disposal option dictates which regulations apply and ultimately the testing requirements. It is important that the appropriate rules (both state and federal) are consulted when establishing your target analyte list.

It is important that the appropriate rules (both state and federal) are consulted when establishing your target analyte list.

Federal monitoring requirements for land application specify testing that measures pollutant concentrations, pathogen reduction, and vector attraction reduction. Disposal of sludge in a monofill or solid waste landfill requires a different testing regime. For example, a facility that land applies its biosolids should analyze its sludge for nutrients and total metals (e.g., arsenic, cadmium, etc.); a facility utilizing composting may sample for nutrients, metals, and salts; and a facility that landfills its sludge may be required to test the sludge by a leaching method such as TCLP extraction. Additional analyses may be required as regulations are changed. Appendix D lists the analyses required for land application of biosolids under federal regulations (40 CFR Part 503) and for the New England states and New York.

In cases where the goal of a sampling program is something other than demonstrating regulatory compliance, the monitoring requirements are less rigid. In this situation operators generally base their testing on operational needs and cost. For example, biosolids managers may choose to perform maturity/stability tests on their compost. These tests may not be required by regulation, but they indicate the quality of the compost as a product and increase its marketability.

Analytical Methods and Protocols

Sewage sludge is a complex mixture composed of organic and inorganic material. This mixture can be heterogeneous and vary over time with respect to its physical, chemical, and biological properties. As discussed in Chapter 4, the character of the wastewater entering a POTW and the type of wastewater and sludge treatment employed largely determine the properties of the solids produced. The variability and complexity of sludge increases the difficulty of sludge analysis. The complex mixture of the sludge matrix can result in significant analytical interference. These interferences can compromise the reliability of analytical data.

To ensure the best possible data quality, it is important to choose appropriate analytical methods. An appropriate method is one that has been evaluated and determined to produce acceptable data in terms of accuracy and precision. Generally, EPA or state regulatory agencies have evaluated analytical methods to determine their acceptability for sludge analysis. Once an analytical protocol is chosen, it is important to use it consistently to enhance data repeatability and comparability.

When compliance is your primary objective, the appropriate analytical method is frequently specified in the applicable regulations. However, when implementing a sampling program, be aware that there could be conflicts between state and federal rules relative to acceptable analytical methodologies. As with analyte lists, care must be taken to ensure that the applicable regulations are consulted and that required analytical protocols are used when necessary. Appendix D lists the target analytes and the corresponding analytical methods required for land application of biosolids as mandated by EPA, the New England states, and New York.

Quality Assurance/Quality Standards and Procedures

A critical phase in the development of data quality objectives is the formulation of a quality assurance/quality control (QA/QC) plan. This phase of determining quality objectives may seem overwhelming; however, environmental laboratories and/or EPA methods have established QA/QC criteria. To evaluate an established QA/QC program or develop a plan, the relevant terms must be understood. Understanding the language of QA/QC will make you a better and more informed consumer of laboratory services.

Definitions

The following terms are commonly associated with QA/QC:

Quality Assurance (QA) – a plan, program, or system developed and instituted to assure that a process or product meets required or desired quality standards. QA equates with process control and sets standards for a process or product. In the case of analytical chemistry, a QA program sets standards for the quality of the analytical results in terms of the accuracy, precision, comparability, and to some extent completeness.

Quality Control (QC) – tools for systematic measurement to determine if the standards set as part of the overall quality assurance program have been met. Blanks, duplicates, calibration checks, and matrix spikes are examples of quality control tools used to assess adherence with quality assurance standards. These tools allow laboratories to evaluate the accuracy and precision of analytical results.

Accuracy – a measure of how closely analytical results match a theoretical true value or known concentration. In other words, it is the extent of agreement between an observed value (sampling result) and the accepted or true value of the parameter being measured. It is standard lab practice to add known amounts of an analyte to a sample and then analyze the sample for that analyte. The result is compared to the known value and expressed as "percent recovery." The closer the percent recovery is to 100 percent, the more accurate the results.

Precision – a measure of the repeatability of a process or analytical procedure. Precision measures the level of agreement or variability among a set of repeated measurements obtained under similar conditions. For example, a sample is chosen for repeat analysis, producing two distinct analytical results. The results are compared to each other mathematically and an absolute or relative deviation is calculated. A small deviation indicates a more precise measurement. The results may not be close to the true value, but they are repeatable within a certain tolerance.

Comparability – the degree to which different methods, data sets, and/or decisions agree or are similar. The concept of comparability can include:

- Reporting analytical results in consistent formats and units, compatible with regulatory requirements.
- Recognition that, when used to measure the same analyte, different analytical procedures can produce significantly different results. For purposes of compliance, sampling plans must conform to regulatory requirements or show that an alternate method produces comparable results.

Completeness – the amount of valid data or results obtained compared with the amount of data planned, generally expressed as a percentage. Here, valid results are defined as analytical data that meet the precision and accuracy data quality objectives established as part of the QA/QC plan. Completeness can be evaluated for a single sample or a data set that has multiple results.

Detection Limits – an important consideration when developing data quality objectives. In general terms, detection limit refers to the lowest concentration that can be reliably detected or reported for a given analyte using a given analytical method. Laboratories report data referring to their detection limits by different names such as minimum detection limit (MDL), reporting detection limit (RDL), and practical quantitation limit (PQL). Each one of these terms has a slightly different meaning, but in each case they refer to the lowest concentration that a lab will report for a given analytical method. In all cases, the detection limit (whether MDL, RDL, or PQL) should be below the specific regulatory limit in order to demonstrate compliance.

In all cases, the detection limit (whether MDL, RDL, or PQL) should be below the specific regulatory limit in order to demonstrate compliance.

Developing a QA/QC Plan

Developing and implementing a QA/QC plan can seem like a daunting task for an operator of a small facility or anyone without previous experience. However, the development process is somewhat simplified because of the availability of helpful resources. EPA-approved analytical protocols, Standard Methods for the Examination of Water and Wastewater, and the American Society of Testing Materials (ASTM) methods all provide recommended appropriate QA/QC methods and mechanisms.

Generally, EPA methods are very prescriptive regarding calibration procedures, numbers of duplicates and spikes, and other QCs that must be performed to document data quality. EPA-approved analytical methods also provide acceptance criteria for required QC efforts. Further, if the lab performing the analyses is certified under the National Environmental Laboratory Accreditation Program (NELAP) then that lab has shown that it is meeting minimum requirements for QA/QC. Again, the key is to ensure that the QA/QC employed by the lab is acceptable according to the rules for which compliance is being sought—if that is the goal of the sampling effort.

Below are general guidelines for required QA/QC that are applicable to the field and laboratory portions of a sampling plan. Please be aware that, particularly for the laboratory, the necessary QA/QC will depend on the analyte and the analytical method being employed. Appendix D lists more detailed information for specific EPA-approved analytical methods commonly used for biosolids analysis.

Field QA/QC

In the field, QA/QC is sometimes overlooked. POTW operators should be aware that data quality objectives and QA/QC start with the collection of the samples. Trip blanks, equipment blanks, replicates, and field duplicates are examples of field QA/QC.

Trip and Equipment Blanks – samples carried into or collected in the field to assess the potential for contamination of samples during the sampling process. Personnel taking samples that will be analyzed for volatile organic compounds should carry a trip blank, which is subject to the same conditions during sampling and transportation as the actual samples. Analytes of interest detected in the **trip blank** are presumed to be extraneous to the actual media being sampled and to originate from a different source. This calls into question results for any contaminant detected both in the trip blank and in the sample(s).

An **equipment blank** is used to evaluate the effectiveness of equipment cleaning procedures and the potential that sampling equipment may be transferring contaminants from one sample to another. An equipment blank should be collected when nondisposable sampling equipment is used. To collect an equipment blank, pour distilled or deionized water into and over cleaned sampling equipment. Allow the water to contact the sampling equipment for a period of time that is similar to the time it would take to conduct the actual sampling procedure. Depending on the analyses to be performed, it may be necessary to collect blanks during every sampling event. If target analytes are detected in the blank, then cleaning procedures or the type of sampling equipment used may need to be reevaluated.

For analytical methods that do not explicitly require trip or equipment blanks, it is generally accepted that the collection of one trip blank and/or equipment blank for every 20 samples collected constitutes good field procedure. If target analytes are detected in the blank, then cleaning procedures or the type of sampling equipment used may need to be reevaluated.

Field Replicates and Duplicates – samples collected to assess the precision of the sampling and analytical procedures as well as to evaluate the variability of the matrix being sampled. Field replicates are samples (two or more) collected from the same source and differentiated by the timing or location of their collection. For example, an operator might collect a grab sample from a belt press for percent-solids analysis and then collect a second grab sample 60 seconds later for the same analysis. These two samples are considered to be replicates. If the first grab sample is divided in half and both halves are analyzed separately for solids content, the two samples formed by dividing the original are considered to be field duplicates. Field duplicates are sometimes known as split samples. Again, one set of field duplicates (or replicates) per 20 samples collected is a generally accepted level of field quality control.

Laboratory QA/QC

As discussed, EPA analytical methods generally specify the necessary QA/QC elements, and environmental laboratories should have robust QA/QC programs based on method requirements. To evaluate the quality of data generated by a lab, you should review the lab's QA/QC manual and evaluate the following:

- Analytical methods used to produce data
- QA/QC standards and procedures
- Detection limits
- Procedures for handling data that do not conform to data quality standards
- Procedures for reporting data that do not conform to data quality standards
- Lab performance based on certification status and lab performance samples

When compliance is your primary objective, it is imperative that you confirm that the laboratory is using the correct analytical methods, QA/QC, and detection limits required by regulations. State and federal regulations commonly specify the analytical methods required to assess compliance with pollutant limits, although they do not typically require specific QA/QC procedures. Regulators may, however, require you to document compliance, which may include documentation of your data quality. Fortunately, published analytical procedures frequently cite achievable detection limits and QA/QC procedures. Again, it is the responsibility of the generator who is procuring laboratory services to review the lab QA/QC plan to ensure that detection limits and QA/QC procedures are consistent with the published methodology and therefore acceptable with regard to demonstrating compliance.

It is the responsibility of the generator who is procuring laboratory services to review the lab QA/QC plan to ensure that detection limits and QA/QC procedures are consistent with the published methodology and therefore acceptable with regard to demonstrating compliance.

Another aspect of evaluating laboratory QA/QC is the need to understand how labs handle data and report results having associated QC (e.g., spikes, duplicates, calibration checks) that do not meet QA standards. Key questions to consider include:

- Does the lab reject data and reanalyze samples for which the attendant QC is unacceptable?
- If data are reported, does the report clearly indicate if data quality is suspect and provide an adequate explanation? For example, some laboratories will report values below their calibration range. However, the data will be qualified to indicate that the reported value is only an estimate of the true value. It is important that labs identify any data that are suspect as a result of a defect or deviation from acceptable QC.

Another avenue for evaluating lab QA/QC, as well as general lab practice, is to ascertain if the lab is involved with any laboratory certification programs such as the NELAP or state certification programs.

Key questions to consider include:

- If the lab holds certification, for which analyses and matrices (e.g., drinking water, wastewater, soil, sludge) is it certified?
- Did the certifying authority conduct an onsite audit?
- Does the certification process include analysis of performance evaluation samples that test the lab's ability to produce accurate data? Participation in an accreditation program provides some assurance that a laboratory has a credible QA/QC program and produces relatively accurate and precise data that can be documented.

Additional guidance for assistance in selecting an environmental laboratory is provided in Appendix E.

Sample Type, Frequency, and Size

The goal of any sampling plan is to collect samples that adequately represent the whole sludge profile. The sampler wants to be able to document that the physical, chemical and biological quality of the sample he or she collects represents the characteristics of the sludge that is used or disposed. As noted already, sludge is a complex, variable mixture whose chemical, physical, and biological properties can be significantly influenced by the type of wastewater treatment and sludge treatment processes used. Sludge complexity and variability increase the difficulty of collecting representative samples. The challenge of any sampling plan is to consider and manage the variables inherent to the sampling process in order to produce representative samples.

The challenge of any sampling plan is to consider and manage the variables inherent to the sampling process in order to produce representative samples.

If a sample does not truly reflect the characteristics of the sludge from which it was derived, then the test results are not meaningful. The type (grab or composite), frequency, and size of samples to be collected are variables that can be controlled and that influence the representativeness of a sample. Representativeness can be addressed, especially for sludge stockpiles, through random sampling. For continuous processes, representativeness is best controlled through the number, frequency, and size of the samples collected.

Establishing criteria for the variables that produce a representative sample is part of the process of developing data quality objectives. It is also important to establish sampling parameters in advance and to maintain those parameters to enhance data comparability over time. The fact that a single sample meets regulatory standards is a far less compelling demonstration of acceptable sludge quality than years of data that demonstrate the same. The remainder of this section discusses establishing data quality objectives relative to sample type, frequency, and size.

The fact that a single sample meets regulatory standards is a far less compelling demonstration of acceptable sludge quality than years of data that demonstrate the same.

Type of Sample (grab versus composite)

A **grab sample** is a specific quantity of sludge collected at a specific time and location. A single grab sample can only represent sludge quality at the time and place it was collected. Extrapolating the analytical results of a single grab sample to represent an entire

stockpile or continuous production is not valid. Grab sampling gains validity as historical data accumulate. One instantaneous data point may not convincingly establish sludge quality, but a database showing a consistent pattern may accurately depict sludge quality over time. For continuous processes, improving the comparability of the grab sampling data requires that equally sized samples are collected from the same location. The timing of grab sample collection should be somewhat random to reflect temporal changes in the sludge. Samples to be submitted for microbial analyses are normally taken as grab samples, so that the time between sample collection and analysis can be documented. Additional information pertaining to microbial sampling is contained in Appendix F.

A **composite sample** is many grab samples that have been collected and mixed together to form a single sample. Grab samples can be randomly collected from locations where sludge is stored, such as a roll-off container or stockpile. In a continuous process, grab samples are typically collected from the same location at a specific time interval over a given period of time. The size of the sample can be weighted to reflect time elapsed or flow. For example, a greater time or flow interval would require a proportionally larger sample than a shorter time or smaller volume.

Generally, composite sampling is accomplished by collecting samples of equal size. In the case of continuous processes, the time interval between grab samples is typically kept constant. For example, a 24-hour composite could be produced by collecting 100-milliliter (mL) samples every hour from a conveyor moving sludge between dewatering and the hauling vehicle. Data generated from the analysis of a composite sample are only representative of the average sludge quality produced during the time frame over which the sample was collected or of the "batch" that was sampled. As with grab samples, historical data provide the best representation of sludge quality.

In composite sampling, the grab samples that comprise the composite should be completely and thoroughly mixed. During the analysis process only a small portion of the overall sample is taken for analysis. If the composite sample is not thoroughly mixed, the subsample that is removed for analysis may only be representative of a single grab. An exception to the mixing rule would be samples that are collected for volatile organic compound (VOC) analysis. In this case, the mixing process can promote the volatilization of analytes such that the sample collected is no longer representative of the sludge being sampled. For VOC samples, replicate analyses can be performed. If samples are extracted and preserved in methanol, then composite samples can be produced by extracting grab samples together or by creating a composite of the aliquots of extract from grabs that have been extracted separately.

Frequency and Size of Samples

Broadly defined, **frequency** refers to the number of samples collected over a given period of time. For example, according to requirements of the federal Part 503 regulations, POTWs may sample sludge for nine metals from one to twelve times per year, depending on the amount of sludge the facility generates. Regulations generally specify the sampling frequency on an annual basis. **Sample size** refers to the actual amount (weight or volume) of the sample that is collected.

Analytical protocols require minimum sample sizes to ensure analytical accuracy and precision. Laboratories should be consulted well in advance of any actual sample collection activities to ascertain the minimum sample size for each analytical method. Generally, if a

Ideally, a sample is small enough to be easily handled, preserved, and transported, but large enough to represent the material being sampled. A larger sample is generally considered to be more representative than a small sample; however, it is important to balance this need for representation with the need for preservation and portability when determining optimal sample size.

laboratory provides sample containers, these containers hold a sufficient quantity of material to perform the required analysis.

Laboratories should be consulted well in advance of any actual sample collection activities to ascertain the minimum sample size for each analytical method.

Sampling frequency and sample size are interrelated sampling parameters. If samples are collected over the course of a year, this frequency equates to a larger set of samples for the whole year's production. Likewise, if a single composite sample is collected, the more grab samples that are collected to form the composite, the larger the sample size. It bears repeating that a composite sample may be no more representative than a grab sample if it is not thoroughly mixed. Again, a larger sample is more representative than a smaller sample.

The most commonly asked questions relative to sampling frequency, sample size, and number of grabs per composite are "How often should I collect samples?" and "How many samples should I collect?" Just as sample size and frequency are related, so are these questions. For any particular analyte, a representative sample size or frequency can be determined by evaluating the variability of the historical data for that analyte.

One measure of variability is **standard deviation**. It is measure of how much individual data vary from the overall average of all data. A high standard deviation indicates that data are highly variable and deviate widely from the average. A low standard deviation indicates more consistent results that vary little from the average.

$$S = \sqrt{\frac{\sum \left| \overline{X} - x \right|^2}{N - 1}}$$

To determine the standard deviation of a historical data set use the following formula:

Where:

S = standard deviation

$\overline{X}$ = average or mean of all data points

x = individual data points

N = number of data points in the set

$\sum \left| \overline{X} - x \right|^2$ = sum of square of the difference between the mean and each individual data point

Most spreadsheet applications will automatically calculate standard deviation. After calculating the mean and standard deviation, if the sum of the mean and the standard deviation exceed the regulatory limit for the analyte in

Most spreadsheet applications will automatically calculate standard deviation.

question, then more samples or more frequent sampling is warranted. It could also indicate inadequate analytical precision. If a facility has limited historical data available, a look at sludge-quality data for similarly sized facilities with comparable industrial bases may be helpful. A more definitive method for calculating sampling frequency or the appropriate number of grabs for a composite sample is described in EPA's *An Addendum to the POTW Sludge Sampling and Analysis Guidance Document*, May 1992. The number of samples is calculated as follows:

If a facility has limited historical data available, a look at sludge-quality data for similarly sized facilities with comparable industrial bases may be helpful.

$$N = \frac{T^2 S^2}{\left(RL - \overline{X}\right)^2}$$

Where:

N = the minimum samples to characterize sludge

T = value of Student's t for the appropriate number of historical data points at 90% confidence level – *See Appendix G*

S = standard deviation

RL = the regulatory limit for the analyte in question

$\overline{X}$ = mean of the historical data

Other Factors to Consider

The size of your facility (influent flow) and the amount of sludge generated are factors to consider when determining sampling frequency. EPA regulations recognize this by requiring more frequent sampling at larger generators. In addition to the size of the facility, the amount of mixing and detention time within your facility influence your ability to collect representative samples. POTWs that have long wastewater detention times and extended sludge ages and/or significant mixing in aeration basins or sludge digesters may be able to take fewer grab samples over a shorter time period.

The final use or disposal option for the material sampled should be considered as you establish sampling strategies. Land application increases the potential for environmental exposure to the contaminants that may be in the biosolids. For example, increased sampling and testing frequency is appropriate for biosolids that are land applied to food-chain crops as opposed to materials that are disposed of in a landfill.

If your POTW has industrial users or storm sewers that discharge to wastewater collection systems, you should be aware of potential variability in the loading of pollutants to your facility. Increases in these loadings ultimately affect sludge quality. This variability can be particularly pronounced if the loadings are random or cyclic. For example, storms sewers produce a random loading event every time there is precipitation.

The timing of sampling events should be scheduled to account for the sludge variability that may result from anticipated changes in pollutant loading.

Industrial users that batch-discharge their wastewater may produce cyclic loading. The timing of sampling events should be scheduled to account for the sludge variability that may result from anticipated changes in pollutant loading.

Costs of Sampling and Analysis

Although sampling costs and analyses are not generally considered in the development of data quality objectives, if financial resources are insufficient to perform sampling and analysis that will meet the goals of the sampling program, then either additional resources will need to be allocated or the goals of the sampling program will need to be reevaluated. If a sampling program is intended to demonstrate regulatory compliance but is inadequate for the task, unintended legal and financial consequences may result. To help ensure that limited resources are used wisely, you need to evaluate your costs in light of your stated goals and data quality objectives.

To help ensure that limited resources are used wisely, you need to evaluate your costs in light of your stated goals and data quality objectives.

Laboratory Qualifications

Although not listed as a section for discussion in this guidance, laboratory qualifications should be considered, given their obvious potential to affect data quality. Considerations in selecting a laboratory include:

- Qualifications of the staff who will perform the analyses.
- Experience in analyzing sludge according to the required method.
- Adequate, appropriate, and proper implementation of QA/QC procedures.
- References from past and present customers.
- Certification by a laboratory accreditation program, such as NELAP, which includes performance evaluation samples and onsite audits.

The assessment of laboratory qualifications is particularly important when conducting microbiological sampling and analysis because of the complexity of the sludge matrix and the scarcity of qualified labs doing this type of analysis.

An important consideration when selecting a laboratory is their turnaround time for completing the analyses. Sampling and analyses must be completed sufficiently in advance of the biosolids ultimate use or disposal in order to assure that the analytical results are received and compliance verified prior to the use or disposal of the biosolids.

CHAPTER 5 REFERENCES

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.

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge. US EPA, Office of Research and Development, EPA/625/R-92/013. Revised July 2003.

The Volunteers Monitor's Guide to Quality Assurance Project Plans. US EPA, Office of Wetlands, Oceans, and Watersheds, EPA/841-B-96-003. September 1996.

EPA Region 8 Biosolids Management Handbook. 1999. Accessed at: *www.epa.gov/region8/water/biosolids/biosolidsdown/handbook/index.html*

CHAPTER 6
SELECTING AND DESCRIBING SAMPLING POINTS

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

Any sampling plan must specifically and accurately identify and describe all sample collection points. This description should explain how the selected sampling points were chosen to produce a representative sample that meets the goals of the sampling program.

The first step in selecting sampling points or locations for inclusion in a sampling plan is to review the goals of your sampling program. Clearly defined objectives simplify the process of identifying appropriate sampling locations. If demonstrating compliance with state and federal regulation is your primary concern, then appropriate sampling locations are to some extent defined by the regulations. If your sampling is for process control, then logical sampling points may be readily apparent. For example, if you want to document the efficiency of your dewatering equipment, then the best sampling point would be the first accessible point after the dewatered solids leave the dewatering device.

In Chapter 5, sample type, size, and sampling frequency were discussed as important factors for obtaining a representative sample, the primary underlying objective of any sampling plan. Choosing the appropriate sampling location is equally important. When choosing sampling locations, the following factors should be considered:

- *Representativeness*
- *Type of process—batch or continuous*
- *Accessibility*
- *Safety*

Representativeness

In almost all phases of developing a sampling plan, the issue of sample representativeness arises. Your choice of sampling points can certainly affect the representativeness of a sample. For example, if a POTW sludge is dewatered with a belt filter press and then conveyed to a pug mill for lime stabilization, where is the most representative location to collect a sample? Is it the sludge holding tank or thickener? Is it the sludge conveyor? Is it before or after lime stabilization?

The best choice is the location that produces a representative sample that best meets the stated goals of the sampling plan. If the POTW land applies its biosolids, then sampling sludge after the pathogen and vector attraction reduction processes produces the most representative sample of the material that will actually be land applied. If an operator wishes to observe changes in sludge quality or track the fate of a specific pollutant during sludge processing, then samples from sludge holding tanks and completely processed sludge should be collected. Compliance sampling generally requires that the sludge be collected at the end of the sludge treatment process in the form in which it will be recycled or disposed.

Compliance sampling generally requires that the sludge be collected at the end of the sludge treatment process in the form in which it will be recycled or disposed.

Type of Process—Batch or Continuous

Choosing appropriate sampling locations also depends on whether your wastewater or sludge is treated in a batch or a continuous process. For example, in a wastewater lagoon, sludge is treated and stored in what amounts to a batch process. Sampling sludge from a lagoon entails collecting a number of grab samples from different areas throughout the lagoon. Biosolids in a stockpile or roll-off container, which can be thought of as a "batch," should be sampled in a similar manner. In all of these situations, a composite sample is produced by collecting a predetermined number of grab samples at random points throughout the batch. For larger areas, such as a lagoon, it is best to establish an imaginary grid system and collect grab samples randomly from within the grid.

Another example of a batch sludge process is dewatering using a plate and frame press. Sludge is pumped into the press until it is full and then the water is removed by compression. After the compression cycle is complete, the press is opened and sludge is scraped from each plate and allowed to fall into a container below. Grab samples are collected at multiple locations from either the press or the container and combined to make a composite sample. The key is that for batch situations, grab samples are collected from multiple locations within the batch.

For continuous processes, multiple grab samples are collected from a single location within the process over time. For example, sludge dewatered in a belt filter press is a continuous process. To sample from a belt filter press, a predetermined number of grab samples are typically collected from the first accessible location after the sludge has passed completely through the press. Generally, this collection location is a point of conveyance between the press and a container or truck in which the sludge will be stored until it is transported to its final destination. If such a procedure is used for the collection of samples for microbial analysis, the samples should be collected over a brief time (less than one hour) and the sample container should be cooled to between 0 and 10 degrees Celsius (° C).

Accessibility

A sampling point must be reasonably accessible to be an effective location. Sampling cannot be performed from a location that cannot be reached. It should be recognized that the best sampling point may not be accessible and that sampling will need to be performed at the next best point of accessibility. Accessibility and safety are related in that a sampling point may be physically accessible, but sampling from that location may present a risk of injury.

When sludge is dewatered with a belt filter press, samples should be collected as the material exits the press. Note the accessibility issues and potential safety hazards present.

Safety

Safety risks must be assessed for each potential sampling location. If there is a risk of injury inherent to a particular sampling location, then consider a safer alternative. The entire sampling process should always emphasize safety. Once you choose a sampling location, identify the potential risks associated with that location, take the appropriate safety precautions, and provide protective equipment.

Safety risks must be assessed for each potential sampling location. If there is a risk of injury inherent to a particular sampling location, then consider a safer alternative.

Typical Sampling Points

POTWs can have a wide variety of configurations in their sludge processing operations, including a number of acceptable methods for sludge stabilization, pathogen reduction, and vector attraction reduction. This process variability means that appropriate sampling points can differ from facility to facility. However, certain sludge treatment processes suggest common sampling locations. Table 6-1 lists typical sampling points associated with common sludge treatment methods.

Table 6-1. TYPICAL SLUDGE SAMPLING POINTS

Sludge Type	Sampling Point
Anaerobically Digested	Taps on the discharge side of positive displacement pumps
Aerobically Digested	1. Taps on discharge lines from pumps 2. If batch digestion, sample directly from digester. **Two cautions:** • If aerated during sampling, air entrains in the sample and VOCs may purge with escaping air. • When aeration is shut off, solids separate rapidly in a well digested sludge.
Thickened Sludges	Taps on the discharge side of positive displacement pumps
Heat Treated	Taps on the discharge side of positive displacement pumps **after** decanting **Two cautions:** • Tendency for solids separation • High temperature of sample can cause problems with certain sampling containers due to cooling and contraction of entrained gases.
Lagoons	Use a "sludge judge" to collect samples from a randomized grid-like pattern, then composite the collected samples.
Dewatered 1. Belt Filter Press, Centrifuge, Vacuum Filter Press 2. Plate and Frame Press 3. Drying Beds	 1. Discharge chutes or conveyors 2. Random locations from the press or random locations from the storage container 3. Random locations from grid system established over the beds
Stockpiles or Storage Containers	Random points (varying the depths and locations) within the stockpile or container
Compost	Random points (varying the depths and locations) within stockpiles of finished compost ready for sale/distribution

CHAPTER 6 REFERENCES

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.

Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge. US EPA, Office of Research and Development, EPA/625/R-92/013. Revised July 2003.

EPA Region 8 Biosolids Management Handbook. 1999. Accessed at: *www.epa.gov/region8/water/biosolids/biosolidsdown/handbook/index.html*

CHAPTER 7
SAMPLE COLLECTION PROCEDURES

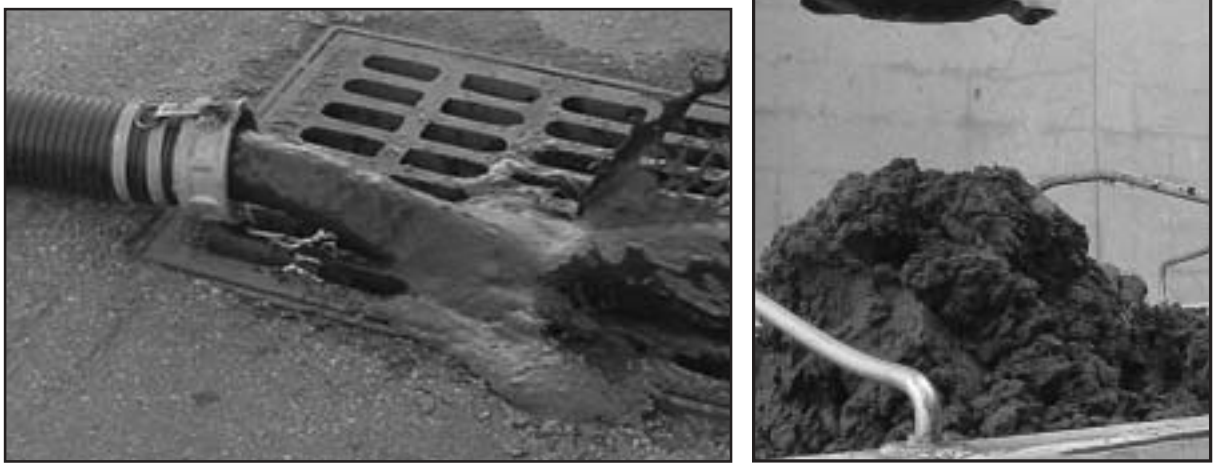
ESSENTIAL ELEMENTS OF A SAMPLING PLAN

- 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.

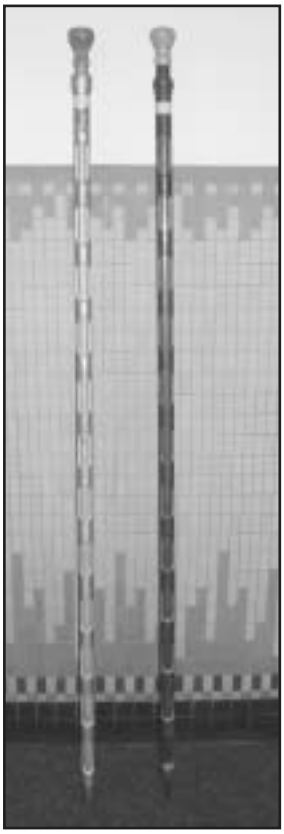
- **Common devices used for sampling solid or semi-solid material**

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.

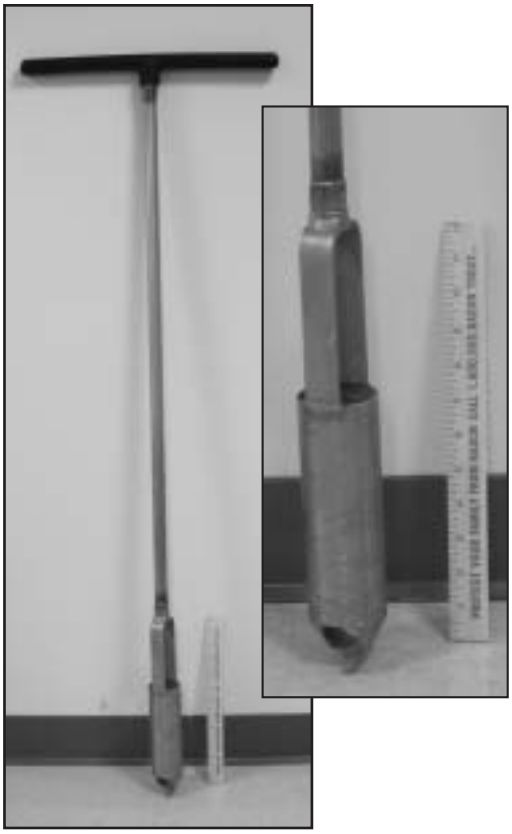
To use this type of sampling device, the thief is pushed into the material to be sampled with the slots opened and oriented upward. Typically, the thief has a pointed or tapered end to facilitate penetration. Once the thief has reached the desired depth, the inner tube is rotated to close the slots and 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)
Tyvek sleeves
Face shield or other appropriate eye protection
Sample handling:
Bucket (accumulate and mix grab samples)
Tongue depressors (transferring sludge)
Small stainless steel hand trowel (transferring or mixing sludge)
Cleaning equipment:
Disposable towels
Soap, such as a low-phosphate laboratory detergent
Scrub brush
Rinse water
Deionized water
Tarp or plastic sheets
Foil or other protective wrap
Sample ID, marking, and labeling:
Labels for sample containers
Custody seals
Pens, pencils, markers
Chain-of-custody form(s)
Field notebook or sample log
Transporting and Preservation:
Sample containers
Cooler
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.

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.

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.

CHAPTER 8
SAMPLE HANDLING 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

A thorough sampling plan documents the procedures employed to preserve and protect the material collected during the sampling event while it is being transported from the field to the laboratory.

Sample Preservation

Preservation refers to sample handling processes aimed at preventing or minimizing chemical or biological activity within the sample after it has been collected. Preservation techniques are different for sludge samples and liquid trip or equipment blank samples. Sludge samples are generally preserved by cooling and maintaining samples at 4° C. Depending on the analytical method, liquid samples may be preserved with the combination of a chemical preservative and chilling to 4° C.

To preserve field and laboratory biosolids samples, cooling to 4° C is (in most cases) the most appropriate method since high-solids sewage sludge cannot be mixed with other preservatives. In addition, laboratory personnel must be notified if chemical preservation is to be done by laboratory staff.

To preserve field and laboratory biosolids samples, cooling to 4° C is (in most cases) the most appropriate method since high-solids sewage sludge cannot be mixed with other preservatives. In addition, laboratory personnel must be notified if chemical preservation is to be done by laboratory staff.

Sample Holding Times

For all environmental samples, the term “holding time” refers to the maximum amount of time that can pass before a sample is analyzed and still obtain valid results. Specific holding times are listed in the details of the particular analytical method used. Holding times can vary from several hours for microbial analysis to several months or longer for metals analysis. For composite samples, the holding time is assumed to commence when the last portion of sampled material has been obtained.

Samples analyzed beyond the maximum holding time generate questionable or invalid results. Therefore, it is essential that analyses are conducted in a timely manner within the specified holding times.

Appendix D lists the specific analyses required by states in the Northeast. It also includes requirements for sample preservation and holding times. Specific analytical methods should always be consulted well in advance of any sampling effort to confirm adherence to method requirements regarding preservation and holding time. State and federal regulatory staff should also be contacted to ensure that proper methods, containers, and preservatives are employed. In addition, the following tables provide general examples of preservation temperatures and maximum holding times, from field collection to analysis, for typical biosolids sample analyses.

Always refer to specific regulations or applicable permits for holding times when sampling for permit compliance.

Always refer to specific regulations or applicable permits for holding times when sampling for permit compliance.

The information in Tables 8-1 and 8-2 is presented to provide general examples only. You should consult the appropriate state and federal regulations and coordinate with the laboratory conducting your analysis to determine specific methods, holding times, preservation, and container requirements that are applicable. This should be done well in advance of any sampling or analysis activities.

Table 8-1. PRESERVATION AND HOLD TIMES FOR ANALYSIS OF BIOSOLIDS SAMPLES

Analyte	Preservation	Maximum Hold Time from Field Collection to Analysis*
Aluminum	Cool to 4° C	6 months
Arsenic	Cool to 4° C	6 months
Barium	Cool to 4° C	6 months
Cadmium	Cool to 4° C	6 months
Chromium	Cool to 4° C	6 months
Copper	Cool to 4° C	6 months
Lead	Cool to 4° C	6 months
Mercury	Cool to 4° C	28 days
Molybdenum	Cool to 4° C	6 months
Nickel	Cool to 4° C	6 months
Selenium	Cool to 4° C	6 months
Silver	Cool to 4° C	6 months

Table 8-1 *continued* **PRESERVATION AND HOLD TIMES FOR ANALYSIS OF BIOSOLIDS SAMPLES**

Analyte	Preservation	Maximum Hold Time from Field Collection to Analysis*
Zinc	Cool to 4° C	6 months
Total Kjeldahl Nitrogen	Cool to 4° C	28 days
Ammonia Nitrogen	Cool to 4° C	28 days
Nitrate Nitrogen	Cool to 4° C	28 days
Total Phosphorus	Cool to 4° C	28 days
Potassium	Cool to 4° C	6 months
Chloride	Cool to 4° C	28 days
Sulfate	Cool to 4° C	28 days
Total Organic Carbon	Cool to 4° C	28 days
Volatile & Semi-volatile Organic Compounds	Cool to 4° C	14 days
Co-planar PCB & Dioxin	Cool to 4° C	1 year
% Total Solids	Cool to 4° C	7 days
% Volatile Solids	Cool to 4° C	7 days
Fecal Coliform	Cool to 4° C	24 hours
Salmonella	Cool to 4° C	24 hours
Enteric Viruses	Cool to between 0 and 10° C	48 hours when between 0 and 10° C
	Freeze sample and hold at -70° C	28 days when cooled to -70° C
Helminth Ova	Cool to between 0 and 10° C	24 hours

** Refer to specific regulations or applicable permits for holding times when sampling for permit compliance.*

Table 8-2. **PRESERVATION AND HOLDING TIMES FOR TCLP ANALYSIS OF BIOSOLIDS SAMPLES**

	Preservation	Maximum Hold Time from Collection to to TCLP Extraction
Organic Compounds	Cool to 4° C	14 days
Mercury	Cool to 4° C	28 days
Metals except Mercury	Cool to 4° C	180 days

Chain of Custody

An integral element of an effective sampling program (documented in a sampling plan) is the use of appropriate chain-of-custody procedures to ensure that laboratory results can be used for compliance, litigation, or enforcement purposes. Chain-of-custody procedures are necessary to ensure the legal integrity of sample materials collected and submitted to a laboratory for analysis. The validity of the test results is enhanced if it can be shown that after the samples were collected they were maintained in a manner that protected them from tampering or interaction with adulterating chemicals. A chain-of-custody form is the written documentation of the security of a sample from the time it is collected to the time it is transferred to the representative of the laboratory that is conducting the analysis. An example of a chain-of-custody form is contained in Appendix J.

A chain-of-custody form is the written documentation of the security of a sample from the time it is collected to the time it is transferred to the representative of the laboratory that is conducting the analysis.

A sample is under your custody if:

- It is in your possession.
- It is in your view, after being in your possession.
- It was in your possession and then locked up to prevent tampering.
- It is in a designated secure area.

Submittal of Samples

Anyone involved in biosolids sampling should, as a standard practice, request chain-of-custody handling of their samples, especially if the results may be used for compliance, enforcement, or other legal purposes. Individuals collecting samples that require chain-of-custody procedures can include regulatory personnel, staff of consulting companies under contract to a regulatory agency, or a biosolids generator/manager taking samples to support a biosolids management program.

Whether samples are hand-carried or delivered by courier to the laboratory, they must be properly preserved, individually sealed, and submitted with a chain-of-custody record. After the laboratory receives the samples, a copy of this record should be returned to the sampling staff. To track samples from collection in the field to receipt of a laboratory report, field sampling personnel may use either a generic or customized chain-of-custody transfer record form.

All too frequently, however, sample containers are not properly protected with custody seals. If you do not seal individual sample containers with custody seals, they will not meet the legal requirements for chain-of-custody protected samples.

If you do not seal individual sample containers with custody seals, they will not meet the legal requirements for chain-of-custody protected samples.

While most laboratories will accept samples that were not maintained and transferred using this procedure, chain-of-custody documentation utilizing custody seals is required in many instances to demonstrate that proper chain-of-custody procedures

were followed in the handling and processing of samples. Samples that are handled and transported without evidence of proper custody may be inadmissible for compliance, enforcement, or other legal purposes.

To further assure the admissibility of biosolids samples for legal purposes, field sampling personnel must observe the following protocols:

- Document in a field notebook all details regarding sampling activities. Documentation must include exact information regarding date, time, location, names of people present, unusual events, field measurements, details of sample storage and security, and transfer of samples to others. If a composite sample is created from multiple grab samples, record the number of grab samples and their relative weighting.
- Use the proper sampling containers, chemicals for sample preservation, coolers, sample labels, chain-of-custody sealing tape, and chain-of-custody record form. Field personnel should also carry with them a listing of acceptable holding times, sampling procedures, and preservation techniques from the laboratory that will conduct the analyses.
- Collect samples according to standard procedures and add a preservative if required. Field personnel needing to break the custody seals at the laboratory to add preservation chemicals should transfer custody to the laboratory after the samples are resealed. Lab personnel must be notified if preservation is to be done at the lab by laboratory staff. The opening and resealing of samples must be documented in field notes and on the chain-of-custody form.

Lab personnel must be notified if preservation is to be done at the lab by laboratory staff.

- After collecting samples, seal the top of each sampling container with a chain-of-custody seal, initial the seal, complete the identifying label, and store and transport the samples in a sealed cooler with ice—if thermal preservation is required. A secure container capable of being sealed is acceptable if thermal preservation is not required. Once all samples are sealed and stored, seal the cooler or container with a custody seal. This seal should be signed by the person who placed the samples inside.
- Use pre-printed, adhesive-backed, chain-of-custody seals (designed for this purpose) on sample containers and coolers. Masking tape, adhesive tape, or common Scotch tape should never be used for chain-of-custody seals because these tapes can be removed without showing evidence of tampering. A failure to use tamper-evident seals may render samples inadmissible for compliance, enforcement, or other legal needs.
- Never leave samples unattended unless they have been locked or secured with initialed custody seals in place.
- Deliver samples to the laboratory supervisor or his/her designee, who will accept the samples and perform the following steps:
  - Verify that correct containers were used and required preservation was performed.
  - Verify that all samples listed on the chain-of-custody record form are accounted for.
  - Verify that all containers are properly sealed, all seals are intact, and the chain-of-custody form and seals are completed correctly.
  - Accept the samples and sign the chain-of-custody record form.
  - Log samples into the laboratory's sample data management system.

– Store samples in a designated locked refrigerator.

– Provide the designated individual with the chain-of-custody record form to file.

Chain-of-Custody Record Form

When using chain-of custody tracking procedures, it is essential that sampling staff complete all items on the chain-of custody record form upon completion of sample collection and prior to submitting the samples to the laboratory. In some jurisdictions, a witness may be required to be present during sampling to satisfy the chain-of-custody requirements of sampling. If a witness is required, they must also sign the form. The form should include the following elements and considerations:

- The project name and number as assigned.
- The sampler(s) (and witness-if required) must sign the form when samples are collected.
- The name of the laboratory performing the analysis.
- The sample location.
- The date and time of sample collection and whether the sample was a composite or grab.
- The description and number of containers, as well as the analyses to be performed.
- The total number of sample containers per location and any remarks regarding the sample.
- The signatures and dates from both parties when custody is transferred from one person to another. If someone other than the person whose signature appears at the top of the form transports the samples to the laboratory, document the transfer of custody on the form.

If the sample is to leave the laboratory for any reason, the sample must be resealed and a chain-of-custody record form reinitiated.

Transportation of Samples

From the time samples are collected, proper handling is critical to sample validity. Exposure to extreme temperatures may compromise collected samples, and testing results may not accurately reflect the true field conditions. Care should be taken to avoid leaving samples in places such as vehicles, postal boxes, and other spaces that could expose them to extreme temperatures. High temperatures can encourage growth of bacteria, which may degrade the organic components in a sample. Freezing is also a concern because it can cause sample containers to break.

Prior to any sample-collection activity, it is very important that the holding times for particular analyses are reviewed and that a suitable method of transporting the samples to the laboratory is selected. For example, samples that have short holding times (e.g., the 6-hour holding time for fecal coliform or salmonella analysis) should not be transported by commercial ground freight service, as they will be received by the laboratory well beyond the allowable holding time. In short, samples must be delivered to the laboratory in the most expedient manner possible following their collection. Note, any material that is identified in the DOT Hazardous Material Table (49 CFR 172.101) must be labeled and transported as prescribed in the table. The three most common methods of delivering samples to the laboratory are:

Hand delivery – This is the most common approach. Unless special arrangements are made, hand delivered samples should be submitted to a laboratory during its normal working hours

and should only be relinquished to the laboratory supervisor or his/her designee.

Overnight-courier or package-delivery service – This method, using commercial carriers such as FedEx or UPS, requires implementation of additional procedures. Thermal preservation must be maintained by packing samples in a sealable cooler with a sufficient volume of ice. (Note: "Blue ice" and most other prepackaged coolants do not cool samples sufficiently.) Unless special arrangements are made, use overnight delivery so that samples arrive before noon during the laboratory's normal workday. It is also highly recommended that sampling events take place early in the week, so samples are received at the laboratory early in their workweek. Samples should not be shipped on a Friday or immediately prior to a holiday unless prior arrangements for receiving the samples have been made with the laboratory.

US Postal Service (USPS) – USPS will not accept for delivery any materials that are classified by the Department of Transportation and postal regulations as hazardous. This restriction precludes USPS delivery of any biosolids samples except for Class A biosolids products. If USPS delivery services are used, the overnight delivery option must be utilized. Be sure that delivery is made directly to the laboratory—not to a centralized "mail room" facility. As with overnight-courier or package-delivery services, samples must be cooled with ice, not with "blue ice" or other prepackaged coolants. Samples should not be shipped on a Friday or immediately prior to a holiday unless prior arrangements for receiving samples have been made with the laboratory.

However samples are delivered, and particularly when using overnight delivery, it is critical that the samples are packed in a manner that ensures that the sample containers are protected from breakage or leakage and that the proper preservation temperature is maintained.

Most laboratories will either reject samples outright or refuse to guarantee their analytical results if samples are received outside of the range of the specified preservation temperature. Generally, samples are required to be chilled to 4° C, with an acceptable range of plus or minus 2° C.

The following is an example of how samples (particularly liquid samples) should be packed for shipping:

Packing Samples for Shipping

1. Insert two large plastic trash bags into the shipping cooler to create a double liner. Immediately before packing the cooler, place an appropriate amount of ice into each of two plastic zipper-lock bags. To prevent leaks, place each ice pack into an additional zipper-lock bag. Seal each zipper-lock bag, expelling as much air as possible, and secure the top with tape.

   Note! Shipping companies may delay sample shipments if leakage occurs. Double liners and zipper-lock bags around ice will prevent leakage and delays.

2. Place a chilled cubitainer—typically a smaller insulated box that fits inside the larger cooler—upright into the center of the lined cooler. Place the two ice packs into the cooler, one on each side of the cubitainer.

3. Place each individual sample container into a zipper-lock bag, expel as much air as possible, seal the bag, and place inside the cubitainer.

4. If you will be monitoring the sample temperature during shipment, place a temperature monitoring device (e.g., extra sample bottle for measuring sample temperature upon receipt at the laboratory, thermometer vial, or Thermochron iButton) inside the cooler. Seal each liner bag by twisting the top of the bag and tying in a knot.

5. Peel the backing off a plastic airbill sleeve and attach the sleeve to the inside of the cooler lid. Alternatively, tape a plastic zipper-lock bag to the lid of the cooler. Sign and date the chain-of-custody form, and place it inside the plastic sleeve or zipper-lock bag on the inside of the cooler lid.

6. Close the cooler lid, seal the horizontal joints with duct or packing tape, and secure the lid with tape by taping the cooler at each end, perpendicular to the seal.

   Note! Shipping companies may delay sample shipments if leakage occurs. Be sure to seal the cooler joints.

7. Peel the backing off of a second airbill sleeve and attach the sleeve to the outside of the cooler lid. Complete the shipping airbill with the laboratory address, billing information, sample weight, and shipping service. Remove the shipper's copy of the airbill, and place the remainder of the airbill inside the plastic sleeve.

8. Ship samples on the day of collection and use a reliable shipping service for next-day delivery. If samples are not shipped on the day of collection, maintain the samples at less than 10° C (but not frozen) by storing in a refrigerator or cooler filled with ice.

9. Notify the laboratory of the sample shipment. Ask the laboratory to contact you to confirm receipt. If the laboratory's receipt of the samples is delayed, use the airbill number to track the sample shipment, utilizing the shipping company's web page or by contacting the shipping company by phone.

At the Laboratory

Once the samples have been received in the laboratory, the supervisor or his/her designee is responsible for their care and custody. That person should be prepared to testify that the samples were in his/her possession or secured in the laboratory at all times—from the moment they were received until the analyses were performed.

CHAPTER 8 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.

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

Sampling Procedures and Protocols for the National Sewage Sludge Survey. US EPA, Office of Water Regulations and Standards (WH-522), Industrial Technology Division. August 1988.

CHAPTER 9
EVALUATION OF COMPLETENESS

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

Once a sampling plan has been developed, samples have been collected and transported to a laboratory for analysis, and results from these analyses have been received, a follow-up step must be conducted to verify that nothing unusual or unanticipated has occurred during the process. The procedures used to accomplish this evaluation of completeness should be documented in the sampling plan.

The evaluation of completeness should include a review of the analytical data received from the laboratory and the effectiveness of the procedures used during the sampling event. It should also discuss whether the initial questions driving the need to collect samples have been adequately addressed. It is important to conduct this evaluation because if there are errors or discrepancies from anticipated results, the samples may need to be retested or additional samples may need to be collected and evaluated.

Data Review

A number of specific items should be reviewed once analytical information has been received from the laboratory performing the sample analysis. The terms "data verification" or "data validation" are often used to describe this process of review. The data review is intended to ensure that the data quality objectives described in Chapter 5 have been met. The following important information should be reviewed:

The data review is intended to ensure that the data quality objectives described in Chapter 5 have been met.

Analytical Methods – Verify that the laboratory performed the proper analyses, based on the methods specified on the chain-of-custody. If the analytical results are to be used to demonstrate compliance with state or federal land application regulations, then it is critical to also verify that the analytical methods correspond to the appropriate methods specified in the state or federal regulations.

Detection Limits – Verify that the analytical results received were analyzed at the appropriate detection limits. Again, when using the analytical results to demonstrate compliance with state or federal land application regulations, the samples must have been analyzed using detection limits that are lower than the applicable concentration limits specified in the regulations.

When using the analytical results to demonstrate compliance with state or federal land application regulations, the samples must have been analyzed using detection limits that are lower than the applicable concentration limits specified in the regulations.

QA/QC – Verify that the appropriate field and laboratory QA/QC samples were analyzed (by the appropriate analytical method and at the correct detection limits). Also, review the results of duplicate, replicate, and spiked samples to be sure that the laboratory is demonstrating the requested precision and accuracy.

Sample Size and Frequency – Because the person reviewing the analytical data may not be the person who collected and submitted the samples to the laboratory, the data reviewer may not be aware of potential changes to the sampling plan, based on field conditions. As a result, the data reviewer should verify that all of the samples that were intended to be collected were in fact collected and that all of the samples submitted to the laboratory were in fact analyzed. For instance, if the sampling plan called for seven grab samples to be collected and submitted for total metals analysis; the data reviewer should verify that seven grab samples were in fact collected and that all seven samples received total metals analysis.

Reporting Format – The data reporting format (particularly as it relates to reporting units) can be critical and should be reviewed to ensure that it is acceptable. If samples were collected to demonstrate compliance or for other regulatory or legal purposes, specific reporting formats and units may be required.

Accuracy of Billing – It is always advisable to review the invoices received from a contracted analytical laboratory and reconcile them with the analytical results received to ensure that there are no overcharges or other billing errors.

Review of Sampling Procedures

After samples have been collected, it is appropriate to review your experiences with specific collection procedures, collection locations, and sample handling and transportation to evaluate the overall effectiveness of the sampling effort. This evaluation and review is a good way to refine and optimize your overall sampling processes. The following review will help you determine process adequacy and allow you to look for opportunities for improvement:

After samples have been collected, it is appropriate to review your experiences with specific collection procedures, collection locations, and sample handling and transportation to evaluate the overall effectiveness of the sampling effort.

1. **Sampling Points** – Key questions for review include:
   - Were the sampling points accessible?
   - Were the samples safely collected from the desired sampling points?
   - Did the sampling points provide representative samples?
   - Are there other potential sampling points that might be better suited for sample collection?

2. **Sampling Procedures** – Key questions to consider include:
   - Was the equipment selected for sample collection adequate for the task?
   - Were the methods of sample collection effective?
   - If a composite sample was collected, was the sludge treatment process amenable to composite sampling?
   - Could the sampling procedures be improved?
3. **Sample Handling** – Key questions to evaluate include:
   - Did the samples arrive at the laboratory intact and at the desired preservation temperature?
   - Were the samples analyzed within the desired holding times or were they unnecessarily delayed due to transportation issues?
   - Were the analyses of the samples unnecessarily delayed at the laboratory?
   - Can the sample handling procedures be improved?
4. **Record-Keeping** – Record-keeping procedures should also be reviewed at this time. Additional information pertaining to record-keeping procedures is presented in Chapter 10.

Overall Review of Your Sampling Program

The period between when your analytical data are received from the laboratory and the analytical results are submitted to a regulatory authority provides you with the opportunity to review the overall effectiveness of your sampling program (as documented in the sampling plan) and of a particular sampling event. Consistent review of the effectiveness of your sampling program ultimately leads to better environmental data.

The following items or issues should be reviewed as you evaluate the effectiveness of your sampling program. *(Note:* These items are biased toward a sampling program designed to demonstrate compliance with state or federal land application regulations.)

- Was the sampling frequency, as defined by regulation, met? For instance, if quarterly sampling is required, were samples collected and analyzed quarterly?
- Were enough samples collected to demonstrate compliance with the appropriate land application regulations?
- Were the samples collected and submitted to the laboratory in sufficient time to allow the analyses to be completed and the results reported before the biosolids sampled were used or disposed?
- Do the analytical data demonstrate compliance with the appropriate land application regulations?
- If sampling was conducted for purposes other than demonstrating environmental compliance, was the initial question or desire for more information sufficiently addressed based on the sample results received?
- Is additional or follow-up sampling necessary or desirable?

CHAPTER 9 REFERENCES

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.

Sampling/Analysis Work Plan Guidance. Maine Department of Environmental Protection, 17 State House Station, Augusta, ME. September 16, 2005.

Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, 3620-BK-DEP2214, Rev. 12/2000. Pennsylvania Department of Environmental Protection, Bureau of Water Quality Protection, Division of Wastewater Management. December 2000.

Sampling Procedures and Protocols for the National Sewage Sludge Survey. US EPA, Office of Water Regulations and Standards (WH-522), Industrial Technology Division. August 1988.

CHAPTER 10
RECORD-KEEPING AND REPORTING REQUIREMENTS

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**

Although record-keeping and reporting are not technically part of the sampling process, any sampling program that ignores these elements will lack documentation of the quality of its sludge. Further, inappropriate or inadequate record-keeping and reporting can be a violation of state and federal regulations. If the primary goal of your sampling program is to demonstrate compliance with applicable regulations, then poor record-keeping and improper reporting can cause serious problems.

Inappropriate or inadequate record-keeping and reporting can be a violation of state and federal regulations

The following information is presented to highlight general record-keeping and reporting requirements under the federal and state regulations governing disposal of sewage sludge. This information pertains to land application and disposal at a sludge-only surface disposal facility (also known as a sludge-only landfill). If your sampling program (as documented in your sampling plan) is being developed for purposes other than sludge disposal, you may still find the information in this chapter useful.

General Considerations

Federal regulations under 40 CFR Part 503 are very specific regarding record-keeping and reporting requirements. The Part 503 rule specifies who must keep records, what records must be kept, and how long records must be retained.

The record-keeping and reporting requirements applicable to EPA are found in 40 CFR 503.17 for POTWs that land-apply materials and in 40 CFR 503.27 for those that dispose material in a sludge-only surface disposal facility. The following three EPA documents provide excellent guidance on record-keeping:

- *Part 503 Implementation Guidance*, EPA 833-R-95-001, Office of Water, October 1995.

- *Preparing Sewage Sludge for Land Application or Surface Disposal: A Guide for Preparers of Sewage Sludge on the Monitoring, Record-keeping, and Reporting Requirements of the Federal Standards for the Use or Disposal of Sewage Sludge,* 40 CFR Part 503, EPA 831B-93-002a, Office of Water, August 1993.
- *Land Application of Sewage Sludge: A Guide for Land Appliers on the Requirements of the Federal Standards for the Use or Disposal of Sewage Sludge,* 40 CFR Part 503, EPA 831-B-93-002b, Office of Enforcement and Compliance Assurance, December 1994.

Some states may have monitoring requirements that exceed federal requirements. Additional monitoring generally entails additional record-keeping and reporting. Table 10-1 cites the applicable sections of state regulations that pertain to record-keeping and reporting for the New England states and New York.

The records that need to be retained depend on the quality of the material and its ultimate end use. Some records need to be reported, while others need to be retained to support the information submitted to the permitting authority. Both the POTWs that generate material and those that use or dispose material must keep records. States may have record retention requirements that are more stringent than the federal 503 regulations. In addition to reviewing the federal guidance documents cited in this chapter, you should also contact the appropriate state regulatory agency to confirm the length of time that records must be kept. See Appendix L for regional regulatory contact information for EPA, the New England states, and New York.

Table 10-1. RECORD-KEEPING AND REPORTING REGULATIONS IN NEW ENGLAND AND NEW YORK

State	Land Application	Sludge-Only Disposal	Sludge Treatment and Monitoring
Connecticut	CGS Section 22a-430 of Chapter 446K	CGS Section 22a-430 of Chapter 446K	CGS Section 22a-430 of Chapter 446K
Maine	06-096 CMR 419 6.	06-096 CMR 401 4	06-096 CMR 409 F. G. and H.
Massachusetts	310 CMR 32.20-26, and 60	310 CMR 32.20 and 60	310 CMR 32.60
New Hampshire	Env-Ws 806.11 Env-Ws 806.12	Env-Ws 808.11 Env-Ws 808.12 Env-Ws 809.04	Env-Ws 807.06 Env-Ws 808.11 Env-Ws 808.12 Env-Ws 810.03
New York	Title 6 NYCRR 360-4.7(c)	Title 6 NYCRR 360-2.17(t)	Title 6 NYCRR 360-5.5(e)
Rhode Island	#12-190-008	#12-190-008	#12-190-008
Vermont	Solid Waste Management Rule §§ 6-703, 6-704	Solid Waste Management Rule §§ 6-703, 6-704	Solid Waste Management Rule §§ 6-703, 6-704

Record-Keeping for Sludge Preparers

Record-keeping and reporting should address two distinct activities: sludge preparation and use/disposal. Sludge sampling at a POTW or other sludge treatment facility (e.g., a compost facility) is a process that monitors the preparation of sludge. A sampling plan should therefore focus on the record-keeping and reporting requirements applicable to sludge treatment and preparation. Typical records that should be maintained by a facility preparing sludge for use or disposal include:

- Data documenting pathogen reduction.
- Data documenting vector attraction reduction (VAR).
- Analytical data showing concentrations of regulated contaminants.
- The amount of sludge produced and its final disposition.

Records relating to pathogen reduction generally involve physical measurements (e.g., time, temperature, pH, percent solids) that document that a specific operational standard associated with an anticipated microbial kill has been met. Various processes have been documented for reducing pathogen concentrations in biosolids. Implementing these processes to meet established operational standards provides a degree of confidence that pathogen densities have been reduced to an acceptable level. Examples of records that might be kept to document pathogen reduction are time and pH for class B lime stabilization. With regard to the sale or distribution of class A biosolids, records relating to actual microbial analyses must be maintained.

Record-keeping for VAR is similar to pathogen reduction. If biosolids are treated in a prescribed manner, then vector attractiveness has been reduced. It is necessary to maintain records that demonstrate that the process used meets the prescribed standards. The example used above – maintaining time and pH records for lime stabilization – would also be applicable for VAR.

Record-keeping for analytical data involves retaining and organizing much of the information collected during the sampling process. Some of this information would include:

- Date, time, and sample location.
- Sample type and method of collection.
- Name and contact information of laboratory performing analyses.
- QA/QC procedures.
- Analytical results.

Most data describing the who, what, when, where, and how of your sampling should be retained. Not all these data have to be reported, but the records should be kept. How long the records must be kept depends on the data and the applicable regulatory requirements.

How long the records must be kept depends on the data and the applicable regulatory requirements.

Most regulatory agencies want to know how much sludge a facility has produced and where it went. Regulations typically require preparers to record:

- The total amount of biosolids produced.
- Each location where the preparer's biosolids were used or disposed of.
- The amount received at each use or disposal location.

In all cases, preparers should check state and federal regulations to determine exactly which records they need to maintain. After determining your exact record-keeping needs, it is recommended that you develop data forms, spreadsheets, or databases that include all the records. These forms and record procedures should be included in the facility sampling plan. The sampling plan should also include details on how long records will be maintained or archived.

Reporting Requirements for Sludge Preparers

Considerations sludge preparers should keep in mind regarding reporting include:

- Reporting term and deadline.
- Reporting format.
- Reporting units.
- Data and information that need to be reported.

Part 503 requires sludge preparers to report annually by February 19 on data collected during the previous calendar year. What must be reported depends on how the sludge is treated to reduce pathogens and vector attractiveness and the use or disposal option. Under federal regulations, preparers must declare the methods and standard operating procedures used to treat their sludge and certify that these methods and procedures were followed. Federal guidance should be consulted to assure the certification process is performed properly.

One potential source of error in reporting is the use of incorrect units. EPA requires that sludge amounts be reported in dry metric tons, while states might require reporting in English tons, either wet or dry. Preparers should ensure that the units used in their reports are those required by the regulatory authority.

One potential source of error in reporting is the use of incorrect units.

Finally, some states may provide a specific reporting form. Such a form can make the reporting process simpler for both the sender and recipient of the report because it clearly details what needs to be reported and how. As always, state and federal regulations should be consulted to determine exact reporting requirements. If you are not required to use a specific reporting form, it is advisable to develop your own form to provide consistency. If you maintain a database, you can develop reporting forms that will rapidly produce a report from data entered into the database over the course of the year.

Other Record-Keeping or Reporting Requirements

POTW operators and sludge preparers may have other record-keeping or reporting requirements associated with the land application or disposal of their sludge. Some of these record-keeping and reporting responsibilities may not be directly related to a sludge sampling program. For example, the location of land application sites is subject to recording and reporting under federal regulations, but this is not a consideration in the development of a sampling plan. For POTWs that contract land application or disposal services, the POTW may have to provide analytical data produced by sludge sampling to the contractor. However, it may be the contractor's responsibility to report on the actual land application or disposal activities. Although these other record-keeping and reporting activities are important and require attention, they are not generally considerations in the development and execution of a sampling program.

CHAPTER 10 REFERENCES

Preparing Sewage Sludge for Land Application or Surface Disposal: A Guide for Preparers of Sewage Sludge on the Monitoring, Record-Keeping, and Reporting Requirements of the Federal Standards for the Use or Disposal of Sewage Sludge, 40 CFR Part 503, EPA 831B-93-002a, Office of Water, August 1993.

Land Application of Sewage Sludge: A Guide for Land Appliers on the Requirements of the Federal Standards for the Use or Disposal of Sewage Sludge, 40 CFR Part 503, EPA 831-B-93-002b, Office of Enforcement and Compliance Assurance, December 1994.

Part 503 Implementation Guidance, EPA 833-R-95-001, Office of Water, October 1995

This page is intentionally blank.

Appendix A
Sampling Plan Worksheet

The following worksheet can be used to develop a sampling plan for your facility. Where appropriate, supporting chapters from the main body of this document have been referenced to assist you in developing your own plan. An example of a completed sampling plan is included in Appendix B.

This page is intentionally blank.

SAMPLING PLAN WORKSHEET

(Please provide attachments as needed)

1. General Facility Information:

Facility Name:

Phone: ( )

Street Address:

City: **State:** **Zip**:

2. Contact Person:

Name: **Title:**

Phone: ( )

Street Address:

City: **State:** **Zip:**

---

3. Sampling Plan Objective(s): (For explanation, see Chapter 3)

Provide a statement that describes the goals of the sampling program.

4. Facility Information: (See Chapter 4)

A. *Provide a brief general description of your facility.*
(Example: conventional activated sludge treatment with anaerobic digestion)

B. *Design Flow (MGD):* *Average daily flow (MGD):*
Previous Year's Annual Sludge Production (dry metric tons):

C. *Briefly describe the screening, grit removal, and flow equalization process employed at your facility.*

D. *Describe the industrial pretreatment program, including a list of permitted facilities, the nature of their discharge, and the local limits to which they are subject.*

E. *Describe any treatment processes (such as advanced treatment for nutrient removal) that may affect sludge quality.*

F. *Describe the source and generation of solids. Does the sludge contain primary solids? What is the schedule and rationale for wasting of secondary sludge? How are solids stored? What is the dewatering method and what chemicals are used in the dewatering process?*

G. *How is the sludge treated to achieve pathogen reduction and vector attraction reduction?*

H. *How will the material be used or disposed of?*

5. Data Quality Objectives: (See Chapter 5 and Appendix D)

A. *List the analytes for which testing is required.*

B. *What analytical methods are required?*

C. *Specify the required quality assurance and quality control for each analytical method used.*

D. *What type of samples will be collected (grab or composite)? If a composite sample is collected, how many grab samples will be collected and what will be the interval between grabs? What will be the sample size?*

6. Sampling Points: (See Chapter 6)

Provide a detailed description of all sampling points along with the rationale for their selection.

7. Sample Collection Procedures: (See Chapter 7 and Appendix H)

Please provide a detailed standard operating procedure (SOP) describing the process used for collecting samples. The step-by-step description should include all details pertaining to sample collection, including a description of the cleaning and preparation procedures for sampling equipment and sample containers.

8. Sampling Handling Procedures: (See Chapter 8 and Appendix D, H, and J)

Describe the post-collection sample handling procedures employed to maintain sample integrity. This description should explain how the samples will be preserved and transported, what the appropriate hold-time is for each analysis, and whether a chain-of-custody is required.

9. Evaluation for Completeness: (See Chapter 9)

Describe the process to be used for evaluating the completeness of the sampling effort. Criteria for evaluation might include: Were the goals of the sampling program met? Were data quality objectives achieved? Do the data quality objectives or SOPs need to be revised?

10. Record-Keeping and Reporting: (See Chapter 10)

Provide a description of record-keeping procedures. The description should explain what information will be retained and for how long, how the information will be stored, and what records are required to be reported.

APPENDIX B

EXAMPLE OF A COMPLETED SAMPLING PLAN WORKSHEET

This example of a completed sampling plan worksheet has been included to illustrate the information necessary to document a sampling program for demonstrating compliance with the federal 503 land application requirements. In this example, Attachment A has been developed from information contained in Appendix D and Attachment B has been developed from information in Appendix H.

This page is intentionally blank.

SAMPLING PLAN WORKSHEET

(Please provide attachments as needed)

1. General Facility Information:

Facility Name: Example Wastewater Treatment Facility

Phone: (123) 456-7891

Street Address: 10 Example Road

City: Example **State:** EX **Zip**: 12345

2. Contact Person: John Example

Name: John Example **Title:** Chief Operator

Phone: (123) 456-7891

Street Address: 10 Example Road

City: Example **State:** EX **Zip**: 12345

3. Sampling Plan Objective(s): (For explanation, see Chapter 3)

Provide a statement that describes the goals of the sampling program.

To demonstrate compliance with Federal Part 503 requirements for the land application of sewage sludge.

4. Facility Information: (See Chapter 4)

A. *Provide a brief general description of your facility.*
(Example: conventional activated sludge treatment with anaerobic digestion)

Extended aeration facility

B. *Design Flow (MGD):* 1.0 *Average daily flow (MGD):* 0.75

Previous Year's Annual Sludge Production (dry metric tons): 175

C. *Briefly describe the screening, grit removal, and flow equalization process employed at your facility.*

Pretreatment includes a bar rack and comminutor

D. *Describe the industrial pretreatment program, including a list of permitted facilities, the nature of their discharge, and the local limits to which they are subject.*

Permitted Pretreatment Facilities, Flow, Monitoring, and Local Limits

Permittee's Name	Type of Discharge	Avg. Daily Discharge (gal.)	Monitoring Frequency	Local Limits		
				Analyte	Max (mg/L)	Avg. Daily (mg/L)
Example Plating, Inc.	Electroplating	3,000	Quarterly	Total CN	1.9	1.0
				Cu	4.5	2.7
				Ni	4.1	2.6
				Cr	7.0	4.0
				Zn	4.2	2.6
				Pb	0.6	0.4
				Cd	1.2	0.7
				Total Metals	10.5	6.8
				TTO	4.57	--
Example Foods, Inc.	Food processing	10,000	Quarterly	BOD_5	500	200
				TSS	50	30
				pH (SIU)	9.0	7.5

E. *Describe any treatment processes (such as advanced treatment for nutrient removal) that may affect sludge quality.*

No advanced treatment or special processes that influence sludge quality or quantity

F. *Describe the source and generation of solids. Does the sludge contain primary solids? What is the schedule and rationale for wasting of secondary sludge? How are solids stored? What is the dewatering method and what chemicals are used in the dewatering process?*

The only source of solids is from the final clarifiers. There is no primary sludge. Sludge is wasted to a sludge holding tank on a daily basis. About 10,000 gallons are wasted from the system once per day to maintain a MLSS of 2000 mg/L. in the aeration basins. The sludge holding tank is used to store and thicken sludge prior to dewatering. Sludge is dewatered on a 1.5-meter belt filter press using polymer and lime. Dewatering is done during an 8-hour shift, five days per week.

G. *How is the sludge treated to achieve pathogen reduction and vector attraction reduction?*
To achieve pathogen reduction, this facility uses EPA Class B alternative 2, Processes to Significantly Reduce Pathogens (PSRP), specifically 40 CFR Part 503.32(b)(3) Appendix B, A.5. Lime stabilization. Vector attraction reduction (VAR) is accomplished by VAR alternative 6, specifically 40 CFR Part 503.33(b)(6), lime stabilization.

H. *How will the material be used or disposed of?*

Land application to corn and hay fields

5. Data Quality Objectives: (See Chapter 5 and Appendix D)

A. *List the analytes for which testing is required.*

For land application under Part 503, testing would include the analytes in Attachment A

B. *What analytical methods are required?*

See Attachment A

C. *Specify the required quality assurance and quality control for each analytical method used.*

For the metals shown in Attachment A, SW-846 Method 7000A specifies the QA/QC requirements. These requirements should be met along with any QA/QC requirements imposed by the specific analytical method used.

D. *What type of samples will be collected (grab or composite)? If a composite sample is collected, how many grab samples will be collected and what will be the interval between grabs? What will be the sample size?*

For each sampling event involving metals and nitrogen, 8 grab samples (approximately 200 mL) collected 1 hour apart will be mixed to form a composite sample. Based on federal requirements, this facility must test for metals at least once per year. To ensure compliance and public acceptance, metals samples will be collected quarterly. Also, one duplicate sample will be collected each quarter.

For pH analysis, two grab samples will be collected. One sample will be collected at the beginning of a dewatering shift and the other at the end. Both samples will be analyzed in the following manner. A portion of the sample will be analyzed immediately after collection to establish an initial pH. After two hours, a second portion of the sample will be tested for pH to document that pathogen reduction has been achieved. A final portion will be analyzed 22 hours later to demonstrate VAR.

6. Sampling Points: (See Chapter 6)

Provide a detailed description of all sampling points along with the rationale for their selection.

Samples will be collected as sludge falls from the belt filter press into roll-off container below. Please see pictures below.

Picture 1. Belt Filter Press/Sampling Point

Picture 2. Closer View of Sampling Point

Picture 3. Close-up of Sampling Point

7. Sample Collection Procedures: (See Chapter 7 and Appendix H)

Please provide a detailed standard operating procedure (SOP) describing the process used for collecting samples. The step-by-step description should include all details pertaining to sample collection, including a description of the cleaning and preparation procedures for sampling equipment and sample containers.

See Attachment B

8. Sampling Handling Procedures: (See Chapter 8 and Appendix D, H, and J)

Describe the post-collection sample handling procedures employed to maintain sample integrity. This description should explain how the samples will be preserved and transported, what the appropriate hold-time is for each analysis, and whether a chain-of-custody is required.

See Attachment B

9. Evaluation for Completeness: (See Chapter 9)

Describe the process to be used for evaluating the completeness of the sampling effort. Criteria for evaluation might include: Were the goals of the sampling program met? Were data quality objectives achieved? Do the data quality objectives or SOPs need to be revised?

In January of each year in preparation for reporting under Part 503, the previous year's sampling efforts will be evaluated. The following criteria will be evaluated:

1) Was all required sampling performed?

2) Were data quality objectives met?
   a) analytical protocols
   b) detection limits
   c) reporting units
   d) Analytical QA/QC
   e) Field QA/QC

3) Were required sample handling and preservation procedures followed?
4) Were the SOPs used and followed?
5) Were SOPs adequate or are revisions necessary?
6) Were record-keeping and reporting procedures adequate?

10. Record-Keeping and Reporting: (See Chapter 10)

Provide a description of record-keeping procedures. The description should explain what information will be retained and for how long, how the information will be stored, and what records are required to be reported.

The facility has developed a database that includes the following data:

Sample Tracking for Metals and Nitrogen Samples					
Date of sample	Time of Collection for Each Grab	Time of Collection of Composite	Date Received by Lab	Date Analyzed	Date Reported by Lab
	1. 2. 3. 4. 5. 6. 7. 8.				

Sample Tracking for pH Samples (In-house testing)							
		Initial pH		pH after 2 hrs.		pH after 22 hrs.	
Date of sample	Time of Collection	Time of Analysis	pH	Time of Analysis	pH	Time of Analysis	pH

The following analytical records will be kept.

	Concentration											
Sample Date	As	Cd	Cu	Pb	Hg	Mo	Ni	Se	Zn	TKN	NH_3	NO_3

The above records along with the individual sample custody sheets will be kept for five years.

ATTACHMENT A – FEDERAL SLUDGE ANALYTICAL REQUIREMENTS

Analyte	Required Analytical Methods	Required Detection Limits	Required QA/QC	Sample Handling	
				Container	Preservation
Arsenic	AA Furnace SW-846 Method 7060 AA Gaseous Hydride SW-846 Method 7061 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 20 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Cadmium	AA Furnace SW-846 Method 7131 AA Direct Aspiration SW-846 Method 7130 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 20 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Copper	AA Furnace SW-846 Method 7211 AA Direct Aspiration SW-846 Method 7210 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 200 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time

ATTACHMENT A – FEDERAL SLUDGE ANALYTICAL REQUIREMENTS

Analyte	Required Analytical Methods	Required Detection Limits	Required QA/QC	Sample Handling	
				Container	Preservation
Lead	AA Furnace SW-846 Method 7421 AA Direct Aspiration SW-846 Method 7420 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 100 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Mercury	Cold Vapor (manual) SW-846 Method 7470 SW-846 Method 7471	At a minimum 5 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 28 days hold time
Molybdenum	AA Furnace SW-846 Method 7481 AA Direct Aspiration SW-846 Method 7480 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 35 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Nickel	AA Direct Aspiration SW-846 Method 7520 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 50 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time

ATTACHMENT A – FEDERAL SLUDGE ANALYTICAL REQUIREMENTS

Analyte	Required Analytical Methods	Required Detection Limits	Required QA/QC	Sample Handling	
				Container	Preservation
Selenium	AA Furnace SW-846 Method 7740 AA Gaseous Hydride SW-846 Method 7741 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 10 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Zinc	AA Direct Aspiration SW-846 Method 7950 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 100 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
pH	EPA-9045 SM-4500-H^+	Not applicable	Per method used	Plastic or glass	4º C 24-hours
Total Kjeldahl Nitrogen (TKN)	SM-4500-N_{org} EPA-351.3	Not applicable	Per method used	Plastic or glass	4º C 28-day hold time
Ammonia Nitrogen (NH_3-N)	SM-4500-NH3 SW-846 Method 9200	Not applicable	Per method used	Plastic or glass	4º C 28-day hold time
Nitrate Nitrogen (NO_3-N)	SM-4500-	Not applicable	Per method used	Plastic or glass	4º C 28-day hold time

ATTACHMENT B – SLUDGE SAMPLING PROCEDURE

1. A week to several days prior to the proposed sampling, confirm or schedule sludge processing (dewatering and treatment) to ensure that sludge in the appropriate form (liquid versus dewatered, untreated cake versus treated biosolids) is available for sampling at the proposed date, time, and sampling point.

2. A week to several days prior to the proposed sampling date, schedule/confirm that contract lab performing the analyses is ready and willing to accept samples on the proposed sampling date.

3. At least one day before collecting samples, assemble the equipment necessary to accomplish the proposed sampling. Ensure that all equipment is clean and in good working order (see attached checklist and cleaning procedure).

4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.

5. After arrival in the dewatering room, evaluate dewatering operations. Any observable deviations from normal operation should be noted prior to collecting samples.

6. Put on nitrile gloves and any other required/desired personal safety equipment.

For Metals and Nitrogen:

7. To collect a composite sample for metals, TKN, NH_3, and NO_3 analyses, take the first of 8 grab samples from the belt filter press as biosolids fall into the roll-off container. All grab samples should be collected using a 500 mL Teflon beaker and a stainless steel trowel, and should be approximately 200 mL in volume. After collecting each grab sample, place the sample in the stainless steel bucket and record the time of collection. Wait one hour and collect the next grab sample. Repeat the process until all eight grab samples are collected. Between collecting of grab samples, the previously collected material should be kept cool (at or around 4 degrees Celsius). Ensure that any required or planned field duplicates or blanks are also collected.

8. Once the last grab sample has been collected, thoroughly mix all material accumulated in the stainless steel bucket using a stainless steel trowel. The goal of the mixing process is to produce a homogeneous sample. After the material is completely mixed, record the current time as the composite sample collection time.

9. After mixing, label all sample containers with the following information:

   a) Sample Identification (ID) Number

   b) Date and time of collection

   c) Sample location

   d) Person collecting sample

   e) Preservative

   f) Required test(s)

10. After labeling, fill each individual sample container with portions of the homogenized sample within the stainless steel bucket.

11. After each sample container is filled, seal it with a signed custody seal and place on ice in a cooler for transportation to the laboratory.

12. Prior to delivering the samples to the lab, complete a chain-of-custody form to document proper sample handling.

13. After sample delivery, clean all equipment according to established procedures and store in a clean, dry area (see below).

For pH sampling:

14. To collect pH samples, collect one grab sample of about 400 mL in a glass beaker at the beginning of a dewatering shift and another grab sample at the end of the shift. A field duplicate should be collected for every twenty samples.

15. Record the time of collection for each sample and label each beaker with the date and collection time.

16. Analyze each sample three times to demonstrate pathogen reduction and vector attraction reduction (VAR). Samples should be stored at 4 degrees Celsius between analyses.

EQUIPMENT CHECKLIST

1) Protective Gear
   a. Nitrile gloves
   b. Tyvek sleeves

2) Sample handling and collection
   a. Stainless steel bucket
   b. 500 mL Teflon beaker
   c. Stainless steel trowel
   d. 500 mL glass beaker (2)

3) Transporting and preservation
   a. Sample containers
      1) For metals and nitrogen: use containers provided by the contract lab.
      2) For pH: use beakers prepared in-house according to established cleaning procedures.
   b. Sample cooler – Obtain sample cooler from contract lab. Ice can be purchased locally.

4) Sample ID and Documentation
   a. Markers and pens
   b. Sample container labels
   c. Custody seals
   d. Chain of custody/sample submittal form
   e. Field notebook/sample log/data sheet

5) Cleaning equipment
   a. Disposable towels
   b. Soap
   c. Scrub brush
   d. Rinse water
   e. Deionized water
   d. 10% hydrochloric acid solution
   e. Rinse water
   f. Deionized water
   g. Aluminum foil or plastic wrap

EQUIPMENT PREPARATION AND CLEANING PROCEDURE

The following cleaning procedure is used to clean all plastic, glass, or stainless steel equipment used to collect sludge samples:

1) Rinse equipment with warm tap water to remove the majority of solids.

2) Using a brush and a low-phosphate lab detergent, scrub the equipment to remove all residues.

3) After scrubbing, rinse the equipment three times with tap water.

4) Next, rinse the equipment with a 10% hydrochloric acid solution - allow at least 30 seconds of contact time.

5) Perform a final rinse, which should be a triple rinse with deionized water.

6) After cleaning, allow the equipment to air-dry. To store, cover beakers and buckets with clean aluminum foil or plastic wrap. Sampling implements should also be wrapped in foil or plastic wrap to keep them clean while in storage.

Note: This cleaning procedure is applicable only when sampling for metals, nitrogen, and pH. To sample for other analytes, especially organic contaminants, these procedures should be modified.

This page is intentionally blank.

Appendix C

Pathogen Reduction and Vector Attraction Reduction (PR/VAR) Methods and Options

The following information describes the methods and options available to demonstrate PR/VAR, as listed in the federal 503 land application regulations. The information for Table C-1 through C-4 was taken from EPA's A Plain English Guide to the EPA Part 503 Biosolids Rule (EPA/832/R-93/003). The information for Table C-5 was taken from EPA's Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge (EPA/625/R-92/013, revised July 2003).

This page is intentionally blank.

Table C-1

Summary of the Six Alternatives for Meeting Class A Pathogen Requirements

In addition to meeting the requirements in one of the six alternatives listed below, the requirements in Table 5-2 must be met for all six Class A alternatives.

Alternative 1: Thermally Treated Biosolids

Biosolids must be subjected to one of four time-temperature regimes.

Alternative 2: Biosolids Treated in a High pH-High Temperature Process

Biosolids must meet specific pH, temperature, and air-drying requirements.

Alternative 3: Biosolids Treated in Other Processes

Demonstrate that the process can reduce enteric viruses and viable helminth ova. Maintain operating conditions used in the demonstration after pathogen reduction demonstration is completed.

Alternative 4: Biosolids Treated in Unknown Processes

Biosolids must be tested for pathogens—*Salmonella* sp. or fecal coliform bacteria, enteric viruses, and viable helminth ova—at the time the biosolids are used or disposed, or, in certain situations, prepared for use or disposal.

Alternative 5: Biosolids Treated in a PFRP

Biosolids must be treated in one of the Processes to Further Reduce Pathogens (PFRP) (see Table 5-4).

Alternative 6: Biosolids Treated in a Process Equivalent to a PFRP

Biosolids must be treated in a process equivalent to one of the PFRPs, as determined by the permitting authority.

Table C-2

Processes to Further Reduce Pathogens (PFRPs) Listed in Appendix B of 40 CFR Part 503

1. Composting

Using either the within-vessel composting method or the static aerated pile composting method, the temperature of the biosolids is maintained at 55°C or higher for 3 days.

Using the windrow composting method, the temperature of the biosolids is maintained at 55°C or higher for 15 days or longer. During the period when the compost is maintained at 55°C or higher, the windrow is turned a minimum of five times.

2. Heat Drying

Biosolids are dried by direct or indirect contact with hot gases to reduce the moisture content of the biosolids to 10 percent or lower. Either the temperature of the biosolids particles exceeds 80°C or the wet bulb temperature of the gas in contact with the biosolids as the biosolids leave the dryer exceeds 80°C.

3. Heat Treatment

Liquid biosolids are heated to a temperature of 180°C or higher for 30 minutes.

4. Thermophilic Aerobic Digestion

Liquid biosolids are agitated with air or oxygen to maintain aerobic conditions, and the mean cell residence time of the biosolids is 10 days at 55° to 60°C.

5. Beta Ray Irradiation

Biosolids are irradiated with beta rays from an accelerator at dosages of at least 1.0 megarad at room temperature (ca. 20°C).

6. Gamma Ray Irradiation

Biosolids are irradiated with gamma rays from certain isotopes, such as Cobalt 60 and Cesium 137, at room temperature (ca. 20°C).

7. Pasteurization

The temperature of the biosolids is maintained at 70°C or higher for 30 minutes or longer.

Table C-3

Summary of the Three Alternatives for Meeting Class B Pathogen Requirements

Alternative 1: The Monitoring of Indicator Organisms

Test for fecal coliform density as an indicator for all pathogens. The geometric mean of seven samples shall be less than 2 million MPNs per gram per total solids or less than 2 million CFUs per gram of total solids at the time of use or disposal.

Alternative 2: Biosolids Treated in a PSRP

Biosolids must be treated in one of the Processes to Significantly Reduce Pathogens (PSRP) (see Table 5-7).

Alternative 3: Biosolids Treated in a Process Equivalent to a PSRP

Biosolids must be treated in a process equivalent to one of the PSRPs, as determined by the permitting authority.

Table C-4

Processes to Further Reduce Pathogens (PFRPs) Listed in Appendix B of 40 CFR Part 503

1. Aerobic Digestion

Biosolids are agitated with air or oxygen to maintain aerobic conditions for a specific mean cell residence time at a specific temperature. Values for the mean cell residence time and temperature shall be between 40 days at 20°C and 60 days at 15°C.

2. Air Drying

Biosolids are dried on sand beds or on paved or unpaved basins. The biosolids dry for a minimum of 3 months. During 2 of the 3 months, the ambient average daily temperature is above 0°C.

3. Anaerobic Digestion

Biosolids are treated in the absence of air for a specific mean cell residence time at a specific temperature. Values for the mean cell residence time and temperature shall be between 15 days at 35°C to 55°C and 60 days at 20°C.

4. Composting

Using either the within-vessel, static aerated pile, or windrow composting methods, the temperature of the biosolids is raised to 40°C or higher and maintained for 5 days. For 4 hours during the 5-day period, the temperature in the compost pile exceeds 55°C.

5. Lime Stabilization

Sufficient lime is added to the biosolids to raise the pH of the biosolids to 12 after 2 hours of contact.

Table C-5

Vector Attraction Reduction Options

Requirement	What is Required?	Most Appropriate For:
Option 1 503.33(b)(1)	At least 38% reduction in volatile solids during sewage sludge treatment	Sewage sludge processed by: Anaerobic biological treatment Aerobic biological treatment
Option 2 503.33(b)(2)	Less than 17% additional volatile solids loss during bench-scale anaerobic batch digestion of the sewage sludge for 40 additional days at 30°C to 37°C (86°F to 99°F)	Only for anaerobically digested sewage sludge that cannot meet the requirements of Option 1
Option 3 503.33(b)(3)	Less than 15% additional volatile solids reduction during bench-scale aerobic batch digestion for 30 additional days at 20°C (68°F)	Only for aerobically digested liquid sewage sludge with 2% or less solids that cannot meet the requirements of Option 1 — e.g., sewage sludges treated in extended aeration plants. Sludges with 2% solids must be diluted
Option 4 503.33(b)(4)	SOUR at 20°C (68°F) is ≤1.5 mg oxygen/hr/g total sewage sludge solids	Liquid sewage sludges from aerobic processes run at temperatures between 10 to 30°C. (should not be used for composted sewage sludges)
Option 5 503.33(b)(5)	Aerobic treatment of the sewage sludge for at least 14 days at over 40°C (104°F) with an average temperature of over 45°C (113°F)	Composted sewage sludge (Options 3 and 4 are likely to be easier to meet for sewage sludges from other aerobic processes)
Option 6 503.33(b)(6)	Addition of sufficient alkali to raise the pH to at least 12 at 25°C (77°F) and maintain a pH ≥12 for 2 hours and a pH≥11.5 for 22 more hours	Alkali-treated sewage sludge (alkaline materials include lime, fly ash, kiln dust, and wood ash)
Option 7 503.33(b)(7)	Percent solids ≥ 75% prior to mixing with other materials	Sewage sludges treated by an aerobic or anaerobic process (i.e., sewage sludges that do not contain unstabilized solids generated in primary wastewater treatment)
Option 8 503.33(b)(8)	Percent solids ≥90% prior to mixing with other materials	Sewage sludges that contain unstabilized solids generated in primary wastewater treatment (e.g., heat-dried sewage sludges)
Option 9 503.33(b)(9)	Sewage sludge is injected into soil so that no significant amount of sewage sludge is present on the land surface 1 hour after injection, except Class A sewage sludge which must be injected within 8 hours after the pathogen reduction process	Sewage sludge applied to the land or placed on a surface disposal site. Domestic septage applied to agricultural land, a forest, or a reclamation site, or placed on a surface disposal site
Option 10 503.33(b)(10)	Sewage sludge is incorporated into the soil within 6 hours after application to land or placement on a surface disposal site, except Class A sewage sludge which must be applied to or placed on the land surface within 8 hours after the pathogen reduction process	Sewage sludge applied to the land or placed on a surface disposal site. Domestic septage applied to agricultural land, forest, or a reclamation site, or placed on a surface disposal site
Option 11 503.33(b)(11)	Sewage sludge placed on a surface disposal site must be covered with soil or other material at the end of each operating day	Sewage sludge or domestic septage placed on a surface disposal site
Option 12 503.33(b)(12)	pH of domestic septage must be raised to ≥12 at 25°C (77°F) by alkali addition and maintained ≥ 12 for 30 minutes without adding more alkali	Domestic septage applied to agricultural land, a forest, or a reclamation site or placed on a surface disposal site

Appendix D

State and Federal Analytical Requirements

The following information contains the analytical requirements for treated sewage sludge that is to be land applied contained in federal regulations and the regulations of the New England states and New York. Where applicable, specific analytes, methods, detection limits, containers, preservation, holding times, and reporting units have been provided

Note: Information contained in this section was provided by each listed state and was current at the time this document was developed. Regulations and approved analytical methods are subject to change. When preparing a sampling plan or collecting samples to demonstrate regulatory compliance, always verify with the appropriate regulatory authority (in advance) that you are using the correct analyte list and analytical methods. Please also note that there is no specific information provided for Connecticut; consult the appropriate state regulatory staff and use the federal table if preparing a biosolids sampling plan for use there.

This page is intentionally blank.

FEDERAL SLUDGE ANALYTICAL REQUIREMENTS

Analyte	Required Analytical Methods	Required Detection Limits	Required QA/QC	Sample Handling	
				Container	Preservation
Arsenic	AA Furnace SW-846 Method 7060 AA Gaseous Hydride SW-846 Method 7061 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 20 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Cadmium	AA Furnace SW-846 Method 7131 AA Direct Aspiration SW-846 Method 7130 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 20 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Copper	AA Furnace SW-846 Method 7211 AA Direct Aspiration SW-846 Method 7210 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 200 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time

FEDERAL SLUDGE ANALYTICAL REQUIREMENTS

Analyte	Required Analytical Methods	Required Detection Limits	Required QA/QC	Sample Handling	
				Container	Preservation
Lead	AA Furnace SW-846 Method 7421 AA Direct Aspiration SW-846 Method 7420 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 100 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Mercury	Cold Vapor (manual) SW-846 Method 7470 SW-846 Method 7471	At a minimum 5 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 28 days hold time
Molybdenum	AA Furnace SW-846 Method 7481 AA Direct Aspiration SW-846 Method 7480 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 35 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Nickel	AA Direct Aspiration SW-846 Method 7520 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 50 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time

ATTACHMENT A – FEDERAL SLUDGE ANALYTICAL REQUIREMENTS

Analyte	Required Analytical Methods	Required Detection Limits	Required QA/QC	Sample Handling	
				Container	Preservation
Selenium	AA Furnace SW-846 Method 7740 AA Gaseous Hydride SW-846 Method 7741 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 10 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
Zinc	AA Direct Aspiration SW-846 Method 7950 Inductively Coupled Plasma SW-846 Method 6010	At a minimum 100 mg/kg, otherwise per lab capability	Per requirements of SW-846 Method 7000A and the specific method used	Plastic or glass	4º C 6 mon. hold time
pH	EPA-9045 SM-4500-H^+	Not applicable	Per method used	Plastic or glass	4º C 24-hours
Total Kjeldahl Nitrogen (TKN)	SM-4500-N_{org} EPA-351.3	Not applicable	Per method used	Plastic or glass	4º C 28-day hold time
Ammonia Nitrogen (NH_3-N)	SM-4500-NH3 SW-846 Method 9200	Not applicable	Per method used	Plastic or glass	4º C 28-day hold time
Nitrate Nitrogen (NO_3-N)	SM-4500-	Not applicable	Per method used	Plastic or glass	4º C 28-day hold time

Massachusetts Analytical Requirements for Land Application

Applicable Regulation: 310 CMR 32.00, Rules and Regulations for Treatment, Disposal, and Transportation of Sewage Sludge

Analyte	CAS #	Required Analytical Methods	Sample Container	Preservation	Reporting Units
Arsenic	7440-38-2	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Cadmium	7440-43-9	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Chromium	7440-47-3	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Copper	7440-50-8	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Lead	7439-92-1	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Mercury	7439-97-6	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Molybdenum	7439-98-7	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Nickel	7440-02-0	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Selenium	7782-49-2	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Zinc	7440-66-6	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Fecal Coliform	na	SM-9221D,E	glass or plastic	cool to 4º C	mpn/g
Solids Total	na	SM-2540G	glass or plastic	cool to 4º C	%
Nitrate/Nitrite	na	SW-4500-NO_3 or SW-846 Method 9210 or EPA 353, 3000 series	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
TKN	na	SM-4500-N_{org} or EPA 351.3	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Ammonia	na	SM-4500-NH_3 or EPA 350	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Total Organic Nitrogen	na	calculation	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Available Phosphoric Acid	na	AOAC	glass or plastic	cool to 4º C	%
Soluble Potash	na	AOAC	glass or plastic	cool to 4º C	%
Specific Conductivity	na	SM-2510B	glass or plastic	cool to 4º C	µmho/cm
pH	na	SM-4500H	glass or plastic	cool to 4º C	SI units
TCLP	na	SW-846, Method 1311	glass, plastic or vials	cool to 4º C	mg/l or ppm
Boron	na	SW-846,6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Total Phosphorous	na	SW-846 or EPA 365	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Potassium	na	SW-846	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
PCB's	na	SW-846, 8082	glass or plastic	cool to 4º C	mg/kg, dry wt.
VOC's	na	SW-846, 8260	glass or plastic	cool to 4º C	mg/kg,dry wt.
SVOC's	na	SW-846, 8270	glass or plastic	cool to 4º C	mg/kg,dry wt.

Maine Analytical Requirements

Parameter	Acceptable Methods	Container	Preservation	Hold Time
Ammonia	undefined in 405.6D SM-4500-NH3 other DEP approved	plastic or glass	Cool 4º C H_2SO_4 pH <2 [aqueous]	28 days
Arsenic	SW-846 Method 7060 SW-846 Method 7061 SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Cadmium	SW-846 Method 7130 SW-846 Method 7131 SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Calcium	SW-846 Method 6010 SW-846 Method 7140 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Calcium Carbonate Equivalents	Calculation from calcium/magnesium results [same as SM 2340B] 2.497 [Ca result] + 4.118 [Mg result]			
Chloride	undefined in 405.6D SW-846 Method 9056 other DEP approved	plastic or glass	none	28 days
Chromium	SW-846 Method 6010 SW-846 Method 6020 SW-846 Method 7190 SW-846 Method 7191 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Copper	SW-846 Method 7210 SW-846 Method 7211 SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months

* *This is Table B-1 taken from the Maine DEP sampling plan guide titled:* Residuals Unit Sampling/Analysis Work Plan Guidance, *September 16, 2005., which should be consulted when preparing a sampling plan for use in Maine.*

Maine Analytical Requirments*				
Parameter	**Acceptable Methods**	**Container**	**Preservation**	**Hold Time**
Iron	SW-846 Method 6010 SW-846 Method 7380 SW-846 Method 7381	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Lead	SW-846 Method 7420 SW-846 Method 7421 SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Magnesium	SW-846 Method 6010 SW-846 Method 7450 other DEP approved	plastic or glass	pH<2 HNO3 [aqueous]	6 months
Mercury	SW-846 Method 7470 SW-846 Method 7471 other DEP approved	plastic or glass	Cool 4º C pH<2 HNO_3 [aqueous]	28 days
Molybdenum	SW-846 Method 7480 SW-846 Method 7481 SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Nickel	SW-846 Method 7520 SW-846 Method 6010 SW-846 Method 6020 SW-846 Method 7521 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Nitrate	undefined in 405.6D SM-4500-NO3 SW-846 Method 9056 other DEP approved	plastic or glass	Cool 4º C H_2SO_4 pH <2 [aqueous]	28 days
Nitrite	undefined in 405.6D SW-846 Method 9056 other DEP approved	plastic or glass	Cool 4º C H_2SO_4 pH <2 [aqueous]	28 days

* *This is Table B-1 taken from the Maine DEP sampling plan guide titled:* Residuals Unit Sampling/Analysis Work Plan Guidance, *September 16, 2005., which should be consulted when preparing a sampling plan for use in Maine.*

Maine Analytical Requirments*

Parameter	Acceptable Methods	Container	Preservation	Hold Time
Percent Dry Solids	undefined in 405.6D SM-2540 G other DEP approved	plastic or glass	Cool 4º C	7 days
pH	undefined in 405.6D SW-846 Method 9045 SM-4500 H+ other DEP approved	plastic or glass	none	24 hours [liquids]
Salt toxicity	electrical conductivity other DEP approved	plastic or glass	none	6 months
Selenium	SW-846 Method 7740 SW-846 Method 7741 SW-846 Method 7051 SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Sodium	undefined in 405.6D SW-846 Method 6010 SW-846 Method 7770 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
TCLP (full suite)	SW-846 Method 1311	glass, PFTE-lined cap	Cool 4º C (for VOC analysis)	14 days (for VOC analysis)
Total Carbon	undefined in 405.6D SM-5310 B SW-846 Method 9060 other DEP approved	amber glass with TFE lined caps	Cool 4º C H_2SO_4 pH <2 [aqueous]	
Total Kjeldahl Nitrogen	undefined in 405.6D SM-4500-Norg EPA 351.3 other DEP approved	plastic or glass	Cool 4º C	28 days

* *This is Table B-1 taken from the Maine DEP sampling plan guide titled:* Residuals Unit Sampling/Analysis Work Plan Guidance, *September 16, 2005., which should be consulted when preparing a sampling plan for use in Maine.*

Maine Analytical Requirments*				
Parameter	**Acceptable Methods**	**Container**	**Preservation**	**Hold Time**
Total Phosphorus	undefined in 405.6D SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Total Potassium	undefined in 405.6D SW-846 Method 6010 SW-846 Method 7610 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months
Total Volatile Solids	undefined in 405.6D SM-2540 G other DEP approved	plastic or glass	Cool 4º C	7 days
Zinc	SW-846 Method 7950 SW-846 Method 6010 SW-846 Method 6020 SW-846 Method 7951 other DEP approved	plastic or glass	pH<2 HNO_3 [aqueous]	6 months

* *This is Table B-1 taken from the Maine DEP sampling plan guide titled:* Residuals Unit Sampling/Analysis Work Plan Guidance, *September 16, 2005., which should be consulted when preparing a sampling plan for use in Maine.*

Additional parameters for sewage sludge generated by POTWs with an average daily flow greater than 2.5 millions of gallons/day; POTWs with pulp and paper, tannery, textile-related or other significant industrial wastewater inputs; POTWs required to enact an Industrial Pretreatment Program according to U.S. EPA regulations 40 CFR Part 403; and sludge or residuals from pulp and paper mills, tanneries, textile mills, and ash generators.

Maine Analytical Requirments*

Parameter	Acceptable Methods	Container	Preservation	Hold Time
Dioxins	EPA 1613 SW-846 Method 8290 other DEP approved	amber glass, PFTE-lined cap	Cool 4º C	30 days
Dioxin TEQs	calculated from Dioxins data as per Chapter 405 Table 405.1 and Table 405.2			
Target SVOCs	SW-846 Methods other DEP approved	glass, PFTE-lined cap	Cool 4º C $Na_2S_2O_3$ [aqueous with residual chlorine]	14 days [solids] 7 days [aqueous]
Target VOCs	SW-846 Methods other DEP approved	glass, PFTE-lined cap	Cool 4º C HCl pH <2 [aqueous] $Na_2S_2O_3$ [aqueous with residual chlorine] Methanol preservation [solids] ***See appropriate preparation method*	14 days
Total PCBs	SW-846 Method 8082 SW-846 Method 8270 other DEP approved	glass, PFTE-lined cap	Cool 4º C $Na_2S_2O_3$ [aqueous with residual chlorine]	14 days [solids] 7 days [aqueous]

* *This is Table B-2 taken from the Maine DEP sampling plan guide titled:* Residuals Unit Sampling/Analysis Work Plan Guidance, *September 16, 2005., which should be consulted when preparing a sampling plan for use in Maine.*

Special tests that may be required.

Maine Analytical Requirments*

Parameter	Methods	Notes
Compost Stability	Aerated Pile Dewars Flask Respiration other DEP approved	See Chapter 405.6.D(2)g(i): temperature monitoring in a compost pile See Chapter 405.6.D(2)g(ii): temperature monitoring in a container ASTM method D5975-96 Standard Test Method for Determining the Stability of Compost by Measuring Oxygen Consumption
Pathogens: Salmonella Fecal Coliform Enteric virus Helminth ova	 SM 9260 D SM 9221 D or E ASTM D 4994-89 EPA 600/1-87-014 other DEP approved	Following treatment by one or more of the pathogen reduction standards, residuals which may contain human pathogens may require compliance testing for one or more of these indicator parameters
Target Pesticides	SW846 8081 other DEP approved	May be required based on a description of the process generating the residual

The Department may require analysis for other parameters that, based on a description of the process generating the residual, may be in the residual in significant concentrations to adversely impact the utilization program.

* *This is Table B-3 taken from the Maine DEP sampling plan guide titled:* Residuals Unit Sampling/Analysis Work Plan Guidance, *September 16, 2005., which should be consulted when preparing a sampling plan for use in Maine.*

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
Dichlorodifluoromethane	75-71-8	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Chloromethane	74-87-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Vinyl chloride	75-01-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Chloroethane	75-00-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Bromomethane	74-83-9	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Trichlorofluoromethane	75-69-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Diethyl ether	60-29-7	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Acetone	67-64-1	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1-Dichloroethene	75-35-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Carbon disulfide	75-15-0	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Methylene chloride	75-09-2	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Methyl-t-butyl ether(MTBE)	1634-04-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
trans-1,2-Dichloroethene	156-60-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1-Dichloroethane	75-34-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
2-Butanone(MEK)	78-93-3	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
2,2-Dichloropropane	590-20-7	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
cis-1,2-Dichloroethene	156-59-2	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Chloroform	67-66-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Tetrahydrofuran(THF)	109-99-9	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Bromochloromethane	74-97-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1,1-Trichloroethane	71-55-6	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1-Dichloropropene	563-58-6	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Carbon tetrachloride	56-23-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2-Dichloroethane	107-06-2	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Benzene	71-43-2	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Trichloroethene	79-01-6	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2-Dichloropropane	78-87-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Bromodichloromethane	75-27-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
Dibromomethane	74-95-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
4-Methyl-2-pentanone (MIBK)	108-10-1	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
cis-1,3-Dichloropropene	10061-01-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Toluene	108-88-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
trans-1,3-Dichloropropene	10061-02-6	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1,2-Trichloroethane	79-00-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
2-Hexanone	591-78-6	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,3-Dichloropropane	142-28-9	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Tetrachloroethene	127-18-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Dibromochloromethane	124-48-1	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2-Dibromoethane	106-93-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Chlorobenzene	108-90-7	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1,1,2-Tetrachloroethane	630-20-6	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Ethylbenzene	100-41-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
m&p-Xylene	108-38-3/106-42-3	10.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
o-Xylene	95-47-6	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Styrene	100-42-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Bromoform	75-25-2	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
iso-Propylbenzene	98-82-8	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,1,2,2-Tetrachloroethane	79-34-5	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2,3-Trichloropropane	96-18-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
n-Propylbenzene	103-65-1	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Bromobenzene	108-86-1	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,3,5-Trimethylbenzene	108-67-8	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
2-Chlorotoluene	95-49-8	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
4-Chlorotoluene	106-43-4	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
tert-Butylbenzene	98-06-6	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
1,2,4-Trimethylbenzene	95-63-6	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
sec-Butylbenzene	135-98-8	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
p-isopropyltoluene	99-87-6	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,3-Dichlorobenzene	541-73-1	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,4-Dichlorobenzene	106-46-7	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
n-Butylbenzene	104-51-8	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2-Dichlorobenzene	95-50-1	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2-Dibromo-3-chloropropane	96-12-8	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2,4-Trichlorobenzene	102-82-1	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Hexachlorobutadiene	87-68-3	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Naphthalene	91-20-3	5.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
1,2,3-Trichlorobenzene	87-61-6	2.0	SW-846, Method 8260	glass	Methanol and cool to 4º C	mg/kg dry wt.
Azobenzene	122-66-7	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,4-Dinitrophenol	51-28-5	12.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,4,6-Trichlorophenol	88-06-2	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,4-Dichlorophenol	120-83-2	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,4-Dimethylphenol	105-67-9	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,4,5-Trichlorophenol	95-95-4	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,4-Dinitrotoluene	121-14-2	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2,6-Dinitrotoluene	606-20-2	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2-Chloronaphthalene	91-59-7	10.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2-Chlorophenol	95-57-8	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2-Methylnaphthalene	91-57-6	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2-Methylphenol	95-48-7	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2-Nitroaniline	88-74-4	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
2-Nitrophenol	88-75-5	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
3,3'-Dichlorobenzidine	91-94-1	4.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
3-Nitroaniline	99-09-2	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
3&4-Methylphenol	106-44-5/ 106-44-5	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4,6-Dinitro-2-methylphenol	534-52-1	12.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4-Bromophenyl-phenylether	101-55-3	10.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4-Chloro-3-methylphenol	59-50-7	10.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4-Chloroaniline	106-47-8	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4-Chlorophenyl-phenylether	7005-72-3	10.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4-Nitroaniline	100-01-6	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
4-Nitrophenol	100-02-7	12.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Acenaphthene	83-32-9	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Acenaphthylene	208-96-8	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Anthracene	120-12-7	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Benzidine	92-87-5	12.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Benzo(a)anthracene	56-55-3	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Benzo(a)pyrene	50-32-8	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Benzo(b)fluoranthene	205-99-2	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Benzo(g,h,i)perylene	191-24-2	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Benzo(k)fluoranthene	207-08-9	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
bis(2-Chloroethoxy)methane	111-91-1	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
bis(2-Chloroethyl)ether	111-44-4	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
bis(2-chloroisopropyl)ether	39638-32-9	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
bis(2-Ethylhexyl)phthalate	117-81-7	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Butylbenzylphthalate	85-68-7	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Carbazole	86-74-8	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Chrysene	218-01-9	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Di-n-butylphthalate	84-74-2	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Di-n-octylphthalate	117-84-0	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Dibenz(a,h)anthracene	53-70-3	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Dibenzofuran	132-64-9	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
Diethylphthalate	84-66-2	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Dimethylphthalate	131-11-3	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Fluoranthene	206-44-0	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Fluorene	86-73-7	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Hexachlorobenzene	118-74-1	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Hexachlorocyclopentadiene	77-47-4	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Hexachloroethane	67-72-1	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Indeno(1,2,3-cd)pyrene	193-39-5	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Isophorone	78-59-1	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
n-Nitroso-di-n-propylamine	621-64-7	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
N-Nitrosodimethylamine	62-75-9	4.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
n-Nitrosodiphenylamine	86-30-6	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Nitrobenzene	98-95-3	2.5	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Pentachlorophenol	87-86-5	4.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Phenanthrene	85-01-8	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Phenol	108-95-2	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Pyrene	129-00-0	5.0	SW-846, Method 8270	glass	cool to 4º C	mg/kg dry wt.
Arsenic	7440-38-2	10	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Cadmium	7440-43-9	1.0	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Chromium	7440-47-3	10	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Copper	7440-50-8	10	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Lead	7439-92-1	11	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Mercury	7439-97-6	0.05	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Molybdenum	7439-98-7	18	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Nickel	7440-02-0	10	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Selenium	7782-49-2	18	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Zinc	7440-66-6	10	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Antimony	7440-36-0	8	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
Beryllium	7440-41-7	0.5	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Silver	7440-22-4	4.0	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Thallium	7440-28-0	10	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Aldrin	309-00-2	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
gamma-BHC	58-89-9	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
alpha-BHC	319-84-6	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
delta-BHC	319-86-8	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
beta-BHC	319-85-7	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Chlordane	57-74-9	0.8	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
4,4'-DDT	50-29-3	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
4,4'-DDE	72-55-9	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
4,4'-DDD	72-54-8	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Endosulfan I	959-98-8	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Endosulfan II	33213-65-9	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Endosulfan Sulfate	1031-07-8	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Endrin	72-20-8	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Endrin Aldehyde	7421-93-4	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Heptachlor	76-44-8	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Heptachlor Epoxide	1024-57-3	0.5	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
Toxaphene	8001-35-2	0.8	SW-846, Method 8081	glass	cool to 4º C	mg/kg dry wt.
PCB-1242	53469-21-9	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1254	11097-69-1	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1221	11104-28-2	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1232	11141-16-5	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1248	12672-29-6	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1260	11096-82-5	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1016	12674-11-2	1.0	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
pH	na	na	SM-4500H	glass or plastic	cool to 4º C	SI units

New Hampshire Analytical Requirements for Land Application

Applicable Regulation: Env-Ws 800, Sludge Management Rules

Analyte	CAS #	Minimum Detection Limit (mg/kg)	Required Analytical Method(s)	Recommended Sample Container	Preservation	Reporting Units
Solids Total	na	na	SM-2540G	glass or plastic	cool to 4º C	%
Nitrate/Nitrite	na	30	SW-4500-NO3 or SW-846 Method 9210 or EPA 353, 3000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
TKN	na	300	SM-4500-Norg or EPA 351.3	glass or plastic	cool to 4º C	mg/kg dry wt.
Ammonia	na	30	SM-4500-NH3 or EPA 350	glass or plastic	cool to 4º C	mg/kg dry wt.
Total Organic Nitrogen	na	na	calculation	glass or plastic	cool to 4º C	mg/kg dry wt.
Potassium	na	15	SM-3500K or SW-846 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Phosphorus	7723-14-0	15	SM-4500-P or EPA 365	glass or plastic	cool to 4º C	mg/kg dry wt.
2,3,7,8-TCDD & 2,3,7,8-TCDF	1746-01-6	5 ppt	EPA 1613A	glass	cool to 4º C	ppt TEQ
Remaining Congeners of 2,3,7,8-TCDD	1746-01-6	5 ppt	EPA 1613A	glass	cool to 4º C	ppt TEQ
Cyanide Total	57-12-5	10	SM-4500-CN	glass or plastic	cool to 4º C	mg/kg dry wt.
Enteric Viruses (if applicable)	na	1 PFU/4g	ASTM Method D4994-89	glass or plastic	cool to 4º C	PFU/4g dry wt.

New York Analytical Requirements for Land Application

Applicable Regulation: NYCRR Part 360, Section 360-5.10

Analyte	CAS #	Required Analytical Methods	Recommended Sample Container	Max. Holding Time/ Preservation
VOLATILE ORGANICS				
Acrolein	107-02-8	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Acrylonitrile	107-13-1	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Benzene	71-43-2	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Bromoform	75-25-2	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Carbon tetrachloride	56-23-5	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Chlorobenzene	108-90-7	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Chlorodibromomethane	124-48-1	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Chloroethane	75-00-3	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
2-chloroethylvinyl ether	110-75-8	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Chloroform	67-66-3	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Dichlorobromomethane	75-27-4	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,1-dichloroethane	75-34-3	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,2-dichloroethane	107-06-2	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Trans-1,2-dichloroethylene	156-60-5	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,1-dichloroethylene	75-35-4	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,2-dichloropropane	78-87-5	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,3-dichloropropene	542-75-6	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Ethylbenzene	100-41-4	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Methyl bromide	74-83-9	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Methyl chloride	74-87-3	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Methylene chloride	75-09-2	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,1,2,2-tetrachloroethane	79-34-5	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Tetrachloroethylene	127-18-4	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Toluene	108-88-3	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,1,1-trichloroethane	71-55-6	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
1,1,2-trichloroethane	79-00-5	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Trichloroethylene	79-01-6	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C
Vinyl chloride	75-01-4	SW-846 Method 8260	glass with Teflon liner	14 days/cool to 4º C

New York Analytical Requirements for Land Application

Applicable Regulation: NYCRR Part 360, Section 360-5.10

Analyte	CAS #	Required Analytical Methods	Recommended Sample Container	Max. Holding Time/ Preservation
SEMIVOLATILE ORGANICS				
4-chloro-3-methylphenol	59-50-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2-chlorophenol	95-57-8	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2,4-dichlorophenol	120-83-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2,4-dimethylphenol	105-67-9	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
4,6-dinitro-2-methylphenol	534-52-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2,4-dinitrophenol	51-28-5	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2-nitrophenol	88-75-5	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
4-nitrophenol	100-02-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Pentachlorophenol	87-86-5	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Phenol	108-95-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2,4,6-trichlorophenol	88-06-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Acenapthene	83-32-9	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Acenaphthylene	208-96-8	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Anthracene	120-12-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Benzidine	92-87-5	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Benzo(a)anthracene	56-55-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Benzo(a)pyrene	50-32-8	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Benzo(b)fluoranthene	205-99-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Benzo(g,h,i)perylene	191-24-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Benzo(k)fluoranthene	207-08-9	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Bis(2-chlorethoxy)methane	111-91-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Bis(2-chloroethyl) ether	111-44-4	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Bis(2-chloroisopropyl) ether	108-60-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Bis(2-ethylhexyl) phthalate	117-81-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
4-bromophenyl phenyl ether	101-55-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Butyl benzyl phthalate	85-68-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2-chloronapthalene	91-58-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
4-chlorophenyl phenyl ether	7005-72-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C

New York Analytical Requirements for Land Application

Applicable Regulation: NYCRR Part 360, Section 360-5.10

Analyte	CAS #	Required Analytical Methods	Recommended Sample Container	Max. Holding Time/ Preservation
SEMIVOLATILE ORGANICS				
Chrysene	218-01-9	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Di-n-butyl phthalate	84-74-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Di-n-Octyl phthalate	117-84-0	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Dibenzo(a,h)anthracene	53-70-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
1,2-dichlorobenzene	95-50-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
1,3-dichlorobenzene	541-73-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
1,4-dichlorobenzene	106-46-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
3,3'-dichlorobenzidine	91-94-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Diethyl phthalate	84-66-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Dimethyl phthalate	131-11-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2,4-dinitrotoluene	121-14-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
2,6-dinitrotoluene	606-20-2	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
1,2-diphenylhydrazine	122-66-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Fluoranthene	206-44-0	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Fluorene	86-73-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Hexachlorobenzene	118-74-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Hexachlorobutadiene	87-68-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Hexachlorocyclopentadiene	77-47-4	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Hexachloroethane	67-72-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Indeno(1,2,3-cd)pyrene	193-39-5	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Isophorone	78-59-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Naphthalene	91-20-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Nitrobenzene	98-95-3	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
N-nitrosodipropylamine	621-64-7	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
N-nitrosodiethylamine	62-75-9	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
N-nitrosodiphenylamine	86-30-6	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Phenanthrene	85-01-8	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
Pyrene	129-00-0	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C
1,2,4-trichlorobenzene	120-82-1	SW-846 Method 8270	amber glass with Teflon liner	14 days/cool to 4º C

New York Analytical Requirements for Land Application

Applicable Regulation: NYCRR Part 360, Section 360-5.10

Analyte	CAS #	Required Analytical Methods	Recommended Sample Container	Max. Holding Time/ Preservation
PESTICIDES/PCBs				
Aldrin	309-00-2	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Alpha-BHC	319-84-6	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Beta-BHC	319-85-7	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Delta-BHC	319-86-8	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Gamma-BHC [Lindane]	58-89-9	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Alpha-chlordane	5103-71-9	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Gamma-chlordane	5103-74-2	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
4,4'-DDD [p,p'-TDE]	72-54-8	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
4,4'-DDE [p,p'-DDX]	72-55-9	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
4,4'-DDT	50-29-3	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Dieldrin	60-57-1	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Alpha-endosulfan	959-98-8	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Beta-endosulfan	33213-65-9	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Endosulfan sulfate	1031-07-8	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Endrin	72-20-8	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Endrin aldehyde	7421-93-4	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Heptachlor	76-44-8	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Heptachlor epoxide	1024-57-3	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1016 (Arochlor 1016)	12674-11-2	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1221 (Arochlor 1221)	11104-28-2	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1232 (Arochlor 1232)	11141-16-5	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1242 (Arochlor 1242)	53469-21-9	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1248 (Arochlor 1248)	12672-29-6	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1254 (Arochlor 1254)	11097-69-1	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
PCB-1260 (Arochlor 1260)	11096-82-5	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C
Toxaphene	8001-35-2	SW-846 Method 8081/8082	amber glass	14 days/cool to 4º C

New York Analytical Requirements for Land Application

Applicable Regulation: NYCRR Part 360, Section 360-5.10

Analyte	CAS #	Required Analytical Methods	Recommended Sample Container	Max. Holding Time/ Preservation
METALS				
Arsenic	7440-38-2	SW-846 Method 6010/7060/7061	glass or plastic	6 months/cool to 4º C
Cadmium	7440-43-9	SW-846 Method 6010/7130/7131	glass or plastic	6 months/cool to 4º C
Chromium	7440-47-3	SW-846 Method 6010/7190/7191	glass or plastic	6 months/cool to 4º C
Copper	7440-50-8	SW-846 Method 6010/7210/7211	glass or plastic	6 months/cool to 4º C
Lead	7439-92-1	SW-846 Method 6010/7420/7421	glass or plastic	6 months/cool to 4º C
Mercury	7439-97-6	SW-846 Method 7470/7471	glass or plastic	28 days/cool to 4º C
Molybdenum	7439-98-7	SW-846 Method 6010/7480/7481	glass or plastic	6 months/cool to 4º C
Nickel	7440-02-0	SW-846 Method 6010/7520	glass or plastic	6 months/cool to 4º C
Selenium	7782-49-2	SW-846 Method 6010/7740/7741	glass or plastic	6 months/cool to 4º C
Zinc	7440-66-6	SW-846 Method 6010/7950/7951	glass or plastic	6 months/cool to 4º C
OTHER PARAMETERS				
pH	na	SW-846 Method 9045	glass or plastic	
Solids Total	na	Standard Method - 2540G	glass or plastic	7 days/cool to 4º C
Nitrate	na	Standard Method - 4500-NO3	glass or plastic	28 days/cool to 4º C
TKN	na	Standard Method - 4500-Norg	glass or plastic	28 days/cool to 4º C
Ammonia	na	Standard Method -4500-NH3	glass or plastic	28 days/cool to 4º C
Potassium	7440-09-7	SW-846 Method 6010/7610	glass or plastic	6 months/cool to 4º C
Phosphorus	7723-14-0	Standard Method - 4500-P	glass or plastic	28 days/cool to 4º C
Fecal Coliform	na	Standard Method - 9221E/9222D	glass or plastic	24 hours/cool to 4º C
Salmonella sp.	na	Standard Method - 9260D.1	glass or plastic	24 hours/cool to 4º C
Viable Helminth Ova	na	EPA/625/R-92/013 Appendix I	glass or plastic	1 month/cool to 4º C
Enteric Viruses	na	ASTM Method D4994-89	glass or plastic	24 hours/cool to 4º C

Rhode Island Analytical Requirements for Land Application

Applicable Regulation: #12-190-008, Rules and Regulations for the Treatment, Disposal, Utilization and Transportation of Sewage Sludge

Analyte	CAS #	Required Analytical Method(s)	Sample Container	Preservation	Reporting Units
Arsenic	7440-38-2	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Cadmium	7440-43-9	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Chromium	7440-47-3	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Copper	7440-50-8	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Lead	7439-92-1	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Mercury	7439-97-6	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Molybdenum	7439-98-7	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Nickel	7440-02-0	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Selenium	7782-49-2	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Zinc	7440-66-6	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Fecal Coliform	na	SM-9221D,E	glass or plastic	cool to 4º C	mpn/g
Solids Total	na	SM-2540G	glass or plastic	cool to 4º C	%
Nitrate/Nitrite	na	SW-4500-NO3 or SW-846 Method 9210 or EPA 353, 3000 series	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
TKN	na	SM-4500-Norg or EPA 351.3	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Ammonia	na	SM-4500-NH3 or EPA 350	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Total Organic Nitrogen	na	calculation	glass or plastic	cool to 4º C	mg/kg dry wt. (%)
Available Phosphoric Acid	na	AOAC	glass or plastic	cool to 4º C	%
Soluble Potash	na	AOAC	glass or plastic	cool to 4º C	%
Specific Conductivity	na	SM-2510B	glass or plastic	cool to 4º C	µmho/cm
pH	na	SM-4500H	glass or plastic	cool to 4º C	SI units
TCLP	na	SW-846, Method 1311	glass, plastic or vials	cool to 4º C	mg/l or ppm

DRAFT Vermont Recommended Analytical Methods for Biosolids Testing

Analyte	CAS #	Required Analytical Method(s)	Sample Container	Preservation	Reporting Units
Total Metals					
Arsenic	7440-38-2	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Cadmium	7440-43-9	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Chromium	7440-47-3	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Copper	7440-50-8	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Lead	7439-92-1	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Mercury	7439-97-6	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Molybdenum	7439-98-7	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Nickel	7440-02-0	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Selenium	7782-49-2	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
Zinc	7440-66-6	SW-846, 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt.
TCLP		SW-846, Method 1311	glass vials	cool to 4º C	mg/l
PCBs					
PCB-1242	53469-21-9	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1254	11097-69-1	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1221	11104-28-2	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1232	11141-16-5	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1248	12672-29-6	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1260	11096-82-5	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
PCB-1016	12674-11-2	SW-846, Method 8082	glass	cool to 4º C	mg/kg dry wt.
Pathogen Indicator*					
Fecal Coliform	na	SM-9221	glass or plastic	cool to 4º C	MPN / g
Salmonella	na	SM-9260	glass or plastic	cool to 4º C	MPN / 4 g

**Please note that Vermont intends to replace the fecal coliform test with Methods 1680 and 1681 and the salmonella test with Method 1682, when those methods are adopted by the federal government.*

DRAFT Vermont Recommended Analytical Methods for Biosolids Testing

Analyte	CAS #	Required Analytical Method(s)	Sample Container	Preservation	Reporting Units
Nutrients					
Nitrate/Nitrite	NO3 1479-76-50 NO2 7697-37-2	SM-4500-NO3 or SW-846 Method 9210 or EPA 353, 3000 series	glass or plastic	cool to 4º C	mg/kg dry wt.(or %)
TKN	na	SM-4500-Norg or EPA 351.3	glass or plastic	cool to 4º C	mg/kg dry wt. (or %)
Ammonia	na	SM-4500-NH3 or EPA 350	glass or plastic	cool to 4º C	mg/kg dry wt. (or %)
Total Organic Nitrogen	na	calculation	glass or plastic	cool to 4º C	mg/kg dry wt. (or %)
Total Phosphorus	7723-14-0	SM-4500-P or EPA 365	glass or plastic	cool to 4º C	mg/kg dry wt. (or %)
Total Potassium	7440-97	SM-3500K or SW-846 6000/7000 series	glass or plastic	cool to 4º C	mg/kg dry wt. (or %)
Other					
pH	na	SM-4500H	glass or plastic	cool to 4º C	SI units
Solids Total	na	SM-2540G	glass or plastic	cool to 4º C	%

This page is intentionally blank.

Appendix E

Questions to Assist with Laboratory Selection

The following information is provided to assist you in selecting a laboratory to conduct your sludge sample analyses. This list includes only some of the potential questions that could be asked. Site-specific concerns may alter the direction of your questions. While it may seem obvious, we nonetheless wish to emphasize that if your questions are not answered to your satisfaction, you should seek the services of a different laboratory.

Source: EPA Region 8 Biosolids Management Handbook (1999) Available at:

http://www.epa.gov/region8/water/biosolids/biosolidsdown/handbook/index.html

This page is intentionally blank.

LAB SELECTION QUESTIONS

General Feasibility

- Does the lab routinely perform the required/requested analyses? Are individuals qualified and do they have written qualifications available?
- Is the lab's turn-around time compatible with your schedule?
- Will the geographical location of the lab cause additional expense (telephone and shipping) and potential difficulty in communication?

Concerns Prior to Sample Collection and Shipment

- Will the lab provide coolers and sample containers?
- What type of sample chain-of-custody is commonly used and will the lab provide chain-of-custody forms prior to shipment?
- What form of shipment is commonly used (Federal Express, UPS, other)? Will the lab pay the shipment cost?
- On what days will someone be available to receive sample shipments (Saturday)?
- What type of sample container should be used and does the lab have any specific packaging requirements?

Costs

- How will I be billed (invoice, pre-pay, other) – NEVER PRE-PAY FOR ANALYSES.
- Are sample containers provided for free or are there additional costs involved?
- What are the sample preparation costs and when and how are they incurred (per sample, per analysis, other)?
- Are there any additional costs involved, which I may not be aware of at this time?
- Can a written estimate be provided and what factors might cause the actual price to differ from the estimate?
- Will QA/QC of my samples involve additional cost?

QA/QC Procedures

- Does the lab have and use any of the following protocols:
  - QA Manual
  - Standard Operating Procedures
  - Sample Custody
  - Traceability to Reference Material
  - QC Checks
  - Data Validation
  - Quality Assessments (Spikes, Duplicates, etc.)
  - Control Charts
  - Other Documentation
  - Periodic QA Audits
- If the lab does not have and/or use the above protocols you should inquire as to the reason. Keep in mind that some of these protocols may be contained within other laboratory procedures or protocols.

Data Processing

After the analyses have been conducted, the lab will provide you with a data package summarizing the analyses. The data package can differ greatly from lab to lab, so the following questions should be asked prior to sample shipment. If any of the information below is not included in the data package, ask the lab to provide it.

- What type of report will I receive? Will narrative descriptions be provided for help in evaluating the data package?
- Will the data be presented on a dry weight basis? IF NOT, REQUIRE IT.
- Will I receive a QA/QC report along with the data package?
- If data qualifiers are present, will a key be provided?
- Will the detection limits for each analysis be provided?
- Will the dates and times of all analyses be reported?
- Will the analytical methods used be included?

Appendix F

Microbial Sampling Considerations

The following are additional critical considerations to be incorporated into any sampling plan or event involving microbial parameters.

This page is intentionally blank.

SLUDGE SAMPLING FOR MICROBIAL ANALYSIS

In a sampling plan, collecting a representative sample is a common objective regardless of the target analytes. Much of the information in this document relates to collecting a representative sludge sample is pertinent to collecting samples for microbial analyses. However, specific microbial parameters may require alterations or accommodations in the sampling plan to ensure the collection of representative samples. EPA provides excellent guidance on sludge sampling in general and microbial sampling in particular in *Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge* (July 2003).

This Appendix highlights and discusses differences between sampling sludge for microbial analysis and sampling for chemical contaminants. Many of the planning steps for developing a sludge sampling program aimed at microbial sampling are no different than any other sampling effort. In fact, many sampling programs will require sampling for both microbial and chemical constituents. The same sampling plan can accommodate both types of sampling, and in fact the planning processes described in Chapters 3 through 6 are applicable for any type of sampling. The planning elements in these chapters are related to sampling goals, facility description, data quality, and sampling points. In addition, the same sampling SOP can generally be used for collection of microbial samples. Although the planning process is the same, the resulting sampling plans may be different depending on the testing to be performed.

Microbial analysis of sludge generally involves fecal coliform, Salmonella, enteric virus, and helminth ova. The most pronounced differences between microbial and chemical sampling involve preparation of sample containers and sampling equipment and hold times. Although proper cleaning of equipment and containers prior to any sampling event is necessary to prevent sample contamination, microbial analysis requires the extra step of sterilization. This is particularly important when analyzing Class A biosolids, which should have no detectable levels of certain pathogens. While it is always advisable to analyze samples as soon after collection as possible, it is particularly important with microbial samples, which have much shorter hold times than chemical analytes.

Preparation of Sample Containers and Sampling Equipment

Chapter 7 contains guidance on the choice and preparation of sampling containers and sampling equipment that is pertinent to microbial sampling. Sample containers for microbial samples should be made of glass, polycarbonate or polypropylene. Pre-sterilized plastic bags (now widely available) are also suitable sample containers. A non-reactive metal, such as stainless steel, probably works best for collection equipment. Glass and Teflon are also acceptable, but may be too fragile or costly.

The general cleaning procedures described in Chapter 7 are adequate for the initial cleaning of containers or equipment. After the initial cleaning, containers and equipment must be sterilized. Sterilization can be accomplished by one of the following methods:

1) Heat in an oven at 170° C for at least 2 hours.

2) Autoclave at 121° C for a minimum of 30 minutes.

3) Soak in a 10 percent bleach solution for a minimum of one minute. Note: If this option is used, the equipment should be rinsed three times with sterile water prior to use.

Prior to heating or autoclaving sampling equipment, it is advisable to wrap it in aluminum foil or a kraft paper to prevent contamination while being stored or transported. When sterilizing glass, polycarbonate or

polypropylene bottles in an autoclave, their tops should be loosened to prevent breakage or deformation. Depending on cost and convenience, pre-sterilized disposable containers and sampling equipment may be an effective option. Finally, it is recommended that sampling containers and equipment for microbial sampling be dedicated to that purpose.

Preservation and Hold Times:

Industry-wide, there has been much confusion over what constitutes proper preservation and the correct maximum hold times for microbial samples. The best reference for preservation and hold times is the specific analytical method that will be used. To prevent growth or decay of microbial populations, hold times should be as short as possible. However, this can be a challenge. For example, few labs are capable of performing enteric virus and helminth ova assays, and samples frequently need to be shipped to laboratories for these analyses. Since shipping complicates preservation and extends hold times, it is imperative that samplers know and plan for the short hold times associated with microbial samples. Below is a table partially reproduced from *Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge* (July 2003), which states maximum hold times using specific preservation methods. It is important to note that maximum holding times and temperatures are method-specific and federal and state regulations should be consulted.

Methods, Preservation, and Maximum Hold Times

Analysis	Method	Preservation	Maximum Hold Time
Enteric Viruses	ASTM(1) Method D 4994-89	–18° C	2 weeks
Fecal coliform	SM(2) Part 9221 E or Part 9222 D	4° C (do not freeze)	24 hours
Salmonella sp.	SM Part 9260D or Kenner and Clark (1974)(3)	4° C (do not freeze)	24 hours
Viable Helminth Ova	Yanko (1987)(4)	4° C (do not freeze)	1 month

1) American Society for Testing and Materials

(2) Standards Methods for the Examination of Water and Wastewater (APHA, 1992)

(3) See Appendix G of *Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge* (July 2003)

(4) See Appendix I of *Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge* (July 2003)

For bacterial and viral analyses, prompt chilling of samples is required to ensure that samples remain representative. If sample analysis will not begin within two hours of collection, place the sample container in an ice water bath (for a minimum of 30 minutes prior to refrigeration) immediately following sample collection. Laboratories should be contacted in advance to schedule analyses and ensure samples are handled in a timely manner upon receipt.

Appendix G

Student's T-Test Calculation

The following example details the steps needed to determine the number of samples to be collected, based on historical analytical results and the statistical student's t-test. It has been adapted from information contained in EPA's *An Addendum to the POTW Sludge Sampling and Analysis Guidance Document,* May 1992.

This page is intentionally blank.

HOW TO DETERMINE THE APPROPRIATE NUMBER OF SAMPLES TO BE COLLECTED

In Chapter 5, the following two equations were provided as a method to determine how many samples should be collected to represent the whole or to determine how many grab samples should be collected to form a composite.

(1)

$$S = \sqrt{\frac{\sum \left| \overline{X} - x \right|^2}{N - 1}}$$

Where:

S = standard deviation

$\overline{X}$ = average or mean of all data points

x = individual data points

N = number of data points in the set

$\sum \left| \overline{X} - x \right|^2$ = sum of square of the difference between the mean and each individual data point

(2)

$$N = \frac{T^2 S^2}{\left(RL - \overline{X}\right)^2}$$

Where:

N = the minimum samples to characterize sludge

T = value of Student's t for the appropriate number of historical data points at 90% confidence level

S = standard deviation

RL = the regulatory limit for the analyte in question

$\overline{X}$ = mean of the historical data

To use this method:

1) Assemble your historical analytical data for the analyte of interest.

2) Calculate the mean or average.

3) Calculate the standard deviation using Equation 1.

4) Determine the regulatory limit for the analyte chosen.

5) Find the Student's T value from Table G-1.

6) Using the mean, standard deviation, regulatory limit, and value of Student's T determined above, calculate the appropriate number of samples by using Equation 2.

Table G-1. VALUES FOR STUDENT'S T AT THE 90% CONFIDENCE LEVEL

Degrees of Freedom (df)	T value at 90% Confidence Level
1	6.314
2	2.920
3	2.353
4	2.132
5	2.015
6	1.943
7	1.895
8	1.860
9	1.833
10	1.812
11	1.796
12	1.782
13	1.771
14	1.761
15	1.753
16	1.746
17	1.740
18	1.734
19	1.729
20	1.725
21	1.721
22	1.717
23	1.714
24	1.711
25	1.708
26	1.706
27	1.703
28	1.701
29	1.699
30	1.697
40	1.684
50	1.676
60	1.671
70	1.667
80	1.664
90	1.662
100	1.660
120	1.658
infinity	1.645

Sample Calculation

Below is a step-by-step example calculation. The objective is to determine the number of composite samples that should be collected during the year to produce statistically valid sludge copper (Cu) concentrations. The following historical Cu data (see Table G-2) will be used.

Table G-2. HISTORICAL COPPER DATA

Date of Sample	Copper Concentration (mg/kg)
7/24/02	480
1/13/03	360
6/11/03	330
5/15/03	135
11/6/03	400
1/7/04	189
4/12/04	140
5/27/04	200
10/26/04	79
1/27/05	140
3/22/05	100
5/27/05	268

Step 1: Calculate the average Cu concentration, add all the concentrations and divide by the number of values:

Date of Sample	Copper Concentration (mg/kg)
7/24/02	480
1/13/03	360
6/11/03	330
5/15/03	135
11/6/03	400
1/7/04	189
4/12/04	140
5/27/04	200
10/26/04	79
1/27/05	140
3/22/05	100
5/27/05	268
TOTAL	2821

Average Copper = 2821 ÷ 12 = 235 (rounded to the nearest whole number)

$$\overline{X} = 235$$

Step 2: Calculate the standard deviation. Fortunately most spreadsheet applications will perform the calculation for you. To perform the process by hand, subtract each individual Cu concentration from the average concentration. Next, square the difference between the average and individual values and sum the squares. See Table G-3 for an example of these calculations. This sum of squared differences can be inserted into the numerator of Equation 1 above.

Table G-3. CALCULATING THE SUM OF SQUARED DIFFERENCES

Date of Sample	Cu Concentration (mg/kg)	$(\overline{X} - x)$	$(\overline{X} - x)^2$
7/24/02	480	-245.00	230400
1/13/03	360	-125.00	129600
6/11/03	330	-95.00	108900
5/15/03	135	100.00	18225
11/6/03	400	-165.00	160000
1/7/04	189	46.00	35721
4/12/04	140	95.00	19600
5/27/04	200	35.00	40000
10/26/04	79	156.00	6241
1/27/05	140	95.00	19600
3/22/05	100	135.00	10000
5/27/05	268	-33.00	71824
SUM	2821		186941

The remaining calculation is as follows:

$$\text{Standard Deviation} = \sqrt{\frac{186941}{12-1}} = 130 \text{ (rounded to the nearest whole number)}$$

Step 3: Based on federal regulations, the ceiling limit for Cu is 4300 mg/kg and the pollutant concentration limit is 1500 mg/kg. In this example, we will assume that the facility wants to show compliance with the lower limit.

Going back to Equation 2, we can see that the average, standard deviation, and regulatory limit have been determined. To use the equation, the final value that must be obtained is Student's T at a 90% confidence level. To find Student's T, use Table G-1. First, find the degrees of freedom by subtracting 1 from the number of historical data points you used to determine the average and standard deviation.

Degrees of Freedom (df) = 12 - 1 = 11

Using Table G-1, locate the Student's T for 11 degrees of freedom (1.796).

Now all the values can be inserted into Equation 2 to obtain the number of grab samples to form a composite.

$$\text{Number of composite samples} = \frac{1.796^2 \, x \, 130^2}{(1500 - 235)^2} = 0.03$$

This calculation indicates that, based on historical data and the current regulatory limit, one composite sample should be sufficient to ensure that regulatory limits are being met. However, facilities must perform the sampling required by state and federal regulations regardless of the results of this calculation. Also, facility operators should be aware that the results of this calculation are heavily influenced by the variability of the historical data and the regulatory limit. For example, if the regulatory limit were 400 mg/kg, the results would indicate that two samples were needed. As a rule of thumb, if the mean of historical data plus the standard deviation is greater than the regulatory limit, then Equation 2 may be helpful in determining the appropriate sampling frequency or number of samples.

This page is intentionally blank.

Appendix H

Example of Sludge Sampling Procedure

The following sludge sampling procedure is an example of a Standard Operating Procedure (SOP) for sample collection, which should be included in all sampling plans. This example SOP also contains an equipment list and a process for cleaning the sampling equipment

This example is for a scenario where eight grab samples are collected from sludge coming off a belt filter press. In this scenario each of the eight grab samples are collected 30 minutes apart. All of the grab samples are then mixed to form a composite sample.

This procedure can be modified as necessary to apply to other sampling locations and scenarios

This page is intentionally blank.

SLUDGE SAMPLING STANDARD OPERATING PROCEDURE

1. A week to several days prior to the proposed sampling, confirm sludge processing (dewatering and treatment) to ensure that sludge in the appropriate form (liquid versus dewatered, untreated cake versus treated biosolids) will be available for sampling at the proposed date, time, and sampling point.

2. A week to several days prior to the proposed sampling date, confirm that the contract laboratory performing the analyses is prepared to accept samples on the proposed sampling date.

3. At least one day before collecting samples, assemble the sampling equipment. Ensure that all equipment is clean and in good working order (See attached checklist and cleaning procedure).

4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.

5. After arrival at the sampling location/sampling point (as determined in the sampling plan), evaluate the operation of the sludge handling train (dewatering, biosolids treatment, etc.). Any observable deviations from normal operation should be noted prior to collecting samples.

6. Put on nitrile gloves and any other required/desired personal safety equipment.

7. Collect the first grab sample of the 8 grab samples that will make up the composite and record the time. Using a 500 mL glass beaker and a stainless steel trowel, collect the sample from the belt filter press as the sludge falls into the roll-off container. The first grab sample and all remaining grabs should be approximately equal in volume (~ 200 mL). Do not forget to collect any required field duplicates or blanks.

8. After the first grab has been collected, it should be placed in a stainless steel bucket. A plastic syringe with the luer lock end removed is used to collect a zero-headspace subsample of about 5 mL in volume from the original grab sample. This subsample should be place in a 40 mL glass vial filled with 10 mL of methanol preservative. This sample should be placed on ice and cooled to 4º C until analyzed according to EPA Method 8260 for volatile organic compounds (VOC).

9. After the first grab sample, another grab sample should be collected every 30 minutes and placed in the stainless steel bucket until all 8 grab samples have been collected. Again, the grab samples should be of approximately equal size (weight or volume). During the time between samples, the stainless steel bucket should be covered and placed on ice or refrigerated. (This is necessary whenever the interval between grab samples is longer than five minutes.) The time of collection of the last grab sample should be recorded.

10. Upon collection of the last grab sample, thoroughly mix all material accumulated in the stainless steel bucket with a stainless steel trowel. The goal of the mixing process is to produce a homogeneous sample.

11. After mixing, label all sample containers with the following information:

    a) Sample identification number (ID)

    b) Date and time of collection

    c) Sample location

    d) Person collecting sample

    e) Preservative

    f) Required test(s)

12. After labeling, fill each sample container with portions of the homogenized material in the stainless steel bucket.

13. After each sample container is filled, seal it with a signed custody seal and place it on ice in a cooler for transportation to the laboratory.

14. Prior to delivering the samples to the lab, complete a chain-of-custody sheet to document proper sample handling.

15. After sample delivery, clean all equipment according to established procedures and store in a clean, dry area.

EQUIPMENT CHECKLIST

1) Protective Gear
   a. Nitrile gloves
   b. Tyvek sleeves

2) Sample handling and collection
   a. Stainless steel bucket
   b. 500 mL glass or Teflon beaker
   c. Stainless steel trowel

3) Transporting and preservation
   a. Sample containers
      1) VOC - 40 mL glass vial with 10 mL of methanol preservative
      2) SVOC – 250 mL, wide-mouth, amber glass jar
      3) Pesticides and PCBs – 250 mL, wide-mouth, amber glass jar
      4) Dioxin – 125 mL, wide-mouth, amber glass jar
      5) Metals and nutrients – 500 mL, wide-mouth, clear glass jar
   b. Sample cooler with ice

4) Sample ID and Documentation
   a. Markers and pens
   b. Sample container labels
   c. Custody seals
   d. Chain of custody/sample submittal form
   e. Field notebook/ sample log/field data sheet

5) Cleaning equipment
   a. Disposable towels
   b. Soap
   c. Scrub brush
   d. Rinse water
   e. Deionized water
   d. 10% hydrochloric acid solution
   e. Rinse water
   f. Deionized water
   g. Aluminum foil or plastic wrap

EQUIPMENT PREPARATION AND CLEANING PROCEDURE

The following cleaning procedure should be used to clean all plastic, glass, or stainless steel equipment used to collect sludge samples:

1) Rinse equipment with warm tap water to remove the majority of solids.

2) Using a brush and a low-phosphate lab detergent, scrub the equipment to remove all residues.

3) After scrubbing, rinse the equipment three times with tap water.

4) Next, rinse the equipment with a 10% hydrochloric acid solution – allow at least 30 seconds of contact time.

5) Perform a final rinse, which should be a triple rinse with deionized water.

6) After cleaning, allow the equipment to air-dry. To store, cover beakers and buckets with clean aluminum foil or plastic wrap. Sampling implements should also be wrapped in foil or plastic wrap to keep them clean while in storage.

Note: **This cleaning procedure is applicable only when sampling for metals, nitrogen, and pH. To sample for other analytes, especially organic contaminants, these procedures should be modified. ASTM D5088 (*Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*) provides detailed guidance on equipment cleaning and decontamination procedures.**

Appendix I

Field Data Sheet

The following is an example of a field data sheet that can be used to record specific details of a sludge sampling event. Ideally, one field data sheet should be completed for every sample collected.

This page is intentionally blank.

FIELD DATA SHEET
BIOSOLIDS SAMPLING

		Refer to Sampling Guide Chapter
Facility Name	_____	2
Sample Location	_____	4, 6
Sample Type	☐ Grab	5
	☐ Composite _____ No. of sub samples	
Sample Collection Equipment	_____	7
Name of Person Collecting Sample	_____	8
Sample Date	_____	8
Weather Conditions	_____	8
Time Sample Collected	_____	8
Time Sample Delivered to Lab	_____	8
Sample Handling Procedures	Sample Size _____	7, 8
	Sample Container _____	7, 8
	Sample Preservation _____	7, 8
	Maximum Hold Time _____	7, 8
	Laboratory Destination _____	7, 8
Sample Documentation	Sample Label _____	7, 8
	Chain of Custody Req'd _____	7, 8

Notes _____

_____

_____

Analytes to Be Tested (units)	☐ Total Solids (%)	5
	☐ pH (S.U.)	
	☐ Total Kjeldahl Nitrogen (%, dry weight)	
	☐ Ammonia Nitrogen (%, dry weight)	
	☐ Nitrate Nitrogen (%, dry weight)	
	☐ Total Phosphorus (%, dry weight)	
	☐ Total Potassium (%, dry weight)	
	☐ Arsenic (mg/kg, dry weight)	
	☐ Cadmium (mg/kg, dry weight)	
	☐ Chromium (mg/kg, dry weight)	
	☐ Copper (mg/kg, dry weight)	
	☐ Lead (mg/kg, dry weight)	
	☐ Mercury (mg/kg, dry weight)	
	☐ Molybdenum (mg/kg, dry weight)	
	☐ Nickel (mg/kg, dry weight)	
	☐ Selenium (mg/kg, dry weight)	
	☐ Zinc (mg/kg, dry weight)	
	☐ VOCs (mg/kg, dry weight)	
	☐ SVOCs (mg/kg, dry weight)	
	☐ Pesticides (mg/kg, dry weight)	
	☐ PCBs (mg/kg, dry weight)	
	☐ TCLP (mg/l or ug/l)	
	☐ Other: _____	

This page is intentionally blank.

Appendix J

Example Chain-of-Custody Form

The following is an example of a chain-of-custody (CoC) form. Chain-of-custody procedures should be employed for all samples collected to demonstrate compliance and any samples that might be used for other regulatory or litigation situations. The example provided reflects samples collected to demonstrate industrial pretreatment procedures, not sludge samples for land application. However, the example was selected because it includes (in great detail) the appropriate information to include on a CoC form.

Source: http://water.ci.lubbock.tx.us/Iwmp/CºCs/ChainofCustodyExample2.pdf

This page is intentionally blank.

EXAMPLE

Chain of Custody

EXAMPLE

CITY OF LUBBOCK WATER UTILITIES LABORATORY
CHAIN OF CUSTODY AND ANALYSIS REQUEST

1 of 1

Report to:	Invoice to:	Project No.	Project Name:
Company: City of Lubbock	Company: SAME		Industrial Monitoring
Address: Water Reclamation	Address:	Sampler's Name (printed) 1	
P O Box 2000		Connie Johnson	
Lubbock TX 79457			
Contact:	Contact:		
Phone: (806) 775-2626	Phone:		
Fax: (806) 775-3246	Fax:		

Results by: Fax results ☐

Sample Source	3 Laboratory Ident. #	4 Date (Jan Feb Mar Apr May Jun Jul Aug Sep Oct Nov Dec 2003 2004 2005)	5 Time	6 Type: Composite	Grab	Blank	7 Container: Glass	HDPE	VOA	Sterile	8 Size	9 Matrix: Water	Sludge	Soil	10 Handling: Sample kept	kept during transport	11 Preservative: HCl	H2SO4	HNO3	NaOH	None	12 Analysis Requested: BOD TSS VSS pH Temp	FOG	Total Metals: Ag As Ba Be Cd Cu Cr Hg Ni Pb Se Zn	13 Field pH	14 Field Temp
2 Craig's Manufacturing	1001	6	10:30	X				X			1L	X			X	X					X	X			7.01	25.0 C
Craig's Manufacturing	1002	7	10:50		X		X				1L	X				X	X						X				
Craig's Manufacturing	1003	6	10:30	X				X			250 ml	X			X	X			X					X			
		7	10:45																								

Analyst:

Remarks: All analyses must be done in accordance with 40 CFR Part 136

15

Sample Condition:	Sampler Relinquished: 16	Date: 10/7/03	Received by: 18	Date:
Cool: Yes ☐ No ☐	Connie Johnson	Time: 11:30		Time:
Intact ☐ 17	Relinquished by:	Date:	Received by:	Date:
Checked By: (Initials)		Time:		Time:
	Relinquished by:	Date:	Received by:	Date:
		Time:		Time:
	☐ Dispose of sample after analysis		Received by:	Date:
	☐ Return residual sample to collector			Time:

CHAIN OF CUSTODY / ANALYSIS REQUEST INSTRUCTIONS

To be completed by the sample collector:
(Note: The number of each step corresponds to an indicated portion of the example Chain-of-Custody on the previous page.)

1. Print sample collector's name (must be legible).
2. Print the sample source description (must be legible).
3. Enter the laboratory ID number (supplied by the laboratory).
4. Enter the date(s) the samples were collected.
5. Enter the time(s) the samples were collected.
6. Mark the appropriate column for the sample type.
7. Mark the appropriate column for the type of container used.
8. Enter the sample size.
9. Mark the appropriate column for the sample matrix.
10. Mark the appropriate column for the sample handling.
11. Mark the appropriate column for the preservative used.
12. Fill in the requested analysis and mark the appropriate column for each sample.
13. Analyze pH samples immediately and enter the results.
14. Analyze the temperature samples immediately and enter the results.
15. Enter any notes about a sample or the analysis.
16. The sample collector signs the block and enters the time and date the samples are relinquished.
17. The sample condition block is filled out and initialed by the laboratory representative receiving the samples.
18. Any other subsequent personnel handling the sample must sign the block in this section (18) of the form.

Appendix K

Glossary

The following sources were used to provide definitions for the terms contained in this section.

- *Guide on Environmental Data Verification and Data Validation.* US EPA. (EPA/240/R-02/004). 2002.
- *The Massachusetts Volunteer Monitor's Guidebook to Quality Assurance Project Plans.* MA DEP. (DWM-CN61.0). 2001.
- *Oxford Pocket Dictionary and Thesaurus.* Oxford University Press. 2002.
- E*nvironmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge.* US EPA, Office of Research and Development, EPA/625/R-92/013. Revised July 2003.

This page is intentionally blank.

Accuracy
A data quality indicator – the extent of agreement between an observed value (sampling result) and the accepted (or true) value of the parameter being measured.

Analyte
A discrete chemical component of a sample to be identified and/or measured through analysis.

Auger
A sampling device, resembling a drill, used for coring into soil-like material.

Average
A measure of central tendency. The average is obtained by adding all the numerical values of a given set of observations and dividing by the total number of observations.

Biosolids
The nutrient-rich organic materials resulting from the treatment of municipal sewage in a wastewater treatment facility. When properly treated and processed, these residuals can be recycled and applied as a fertilizer or soil conditioner to improve and maintain productive soils and stimulate plant growth.

Chain-of-Custody
A process used for routine sample control for regulatory and non-regulatory monitoring; also used as a general term to include sample labels, field logging, field sheets, custody seals, lab receipt and assignment, disposal, and all other aspects of sample handling from collection to ultimate analysis. Chain-of-custody also refers to the document or paper trail showing the proper handling of evidence and its integrity.

Coliwasa
Combined Liquid Waste Sampler. A sampling device used to collect a core sample of free-flowing liquid sludge from lagoons, tanks, pits, and similar containments.

Composite Sample
A composite sample can be either A) a collection of individual samples obtained at regular intervals of time or flow; or B) grab samples collected from various locations within a single mass of material, such as a lagoon or stockpile. Once mixed, the collected material is analyzed to determine the average conditions during the sampling period.

Data Validation
An analyte and sample specific process that extends to evaluation of data beyond method, contractual, and procedural compliance (see data verification) to determine the analytical quality of a specific data set.

Data Verification
The process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against method, procedural, or contractual obligations.

Detection Limit
The lowest concentration or measurement of a target analyte that a given method can reliably ascertain as greater than zero.

Duplicate Sample
Two samples taken generally at the same time from (and representative of) the same sampling point that are carried through all assessment and analytical procedures in an identical manner. Used to measure the precision of field sampling and lab analytical methods.

Equipment Blank
A QA/QC sample used to check specifically for carryover contamination from the reuse of sampling equipment.

Grab Sample
A single finite sample collected at a specific location and time.

Holding Time
The elapsed time from the date and time of sample collection until the sample is analyzed.

Homogenous
A uniform mixture having similar quality or characteristics throughout.

Indicator Organisms
Organisms that have been found to respond to treatment processes and environmental conditions in a manner similar to pathogenic organisms.

Malodor
A bad odor, a stench.

Mean
Another term for the average of a set of data; calculated in the same manner as the average.

NELAP
National Environmental Laboratory Accreditation Program

Pathogen
An organism or substance capable of causing disease. Pathogenic organisms include bacteria, viruses, protozoa, and helminths.

Pathogen Reduction
Treatment processes utilized to reduce sewage sludge pathogen concentrations in order to protect public health. Pathogen reduction is accomplished through

treatment of sewage sludge or through a combination of treatment and restrictions on the land application site that prevent exposure to the pathogens and allow time for environmental conditions to reduce the pathogens to below detectable levels.

PCB
Polychlorinated Biphenyl

Precision
A data quality indicator that measures the level of agreement or variability among a set of repeated measurements, obtained under similar conditions. Precision is usually expressed as a standard deviation in absolute or relative terms.

Preservation
Methods used to retard degradation of chemical analytes or physical properties within samples by inhibiting decomposition by biological action and chemical reactions, and reducing sorption effects. Methods include chemical, acid, or base addition; protection from light; cooling; etc.

Preservative
Generally, a chemical added to a sample to prevent decomposition or chemical reaction.

Putrescible
Subject to rotting, decomposition, or decay.

QA/QC
Quality Assurance/Quality Control

Quality Assurance
An integrated management system designed to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control
The overall system of technical activities designed to measure quality and limit error in a product or service.

Replicate Sample
Two samples taken at different times from the same sampling point that are carried through all assessment and analytical procedures in an identical manner.

Sample
A portion of material collected for chemical analyses or measurement. As a general laboratory practice, a sample is identified by a unique sample number. If a single sample is submitted for a variety of chemical analyses, the number may apply to multiple sample containers.

Sampling Plan
The complete written documentation of an organization's sampling program.

Sampling Point
The specific location within the treatment process where material to be sampled is collected.

Sampling Program
All applicable elements, events, materials, and personnel associated with the collection of samples.

Sludge Judge
A sampling device, similar to a coliwasa, used to collect a core sample of free-flowing liquid sludge.

Standard Deviation
A measure of the range of variation among repeated measurements, used in the determination of precision.

TCLP
Toxicity Characteristic Leaching Procedure

Thief Sampler
Consisting of two slotted concentric tubes, a thief sampler is used to sample granulated or powdered sludges. The sampler is pushed into the material to be sampled, the inner tube is rotated to close the sampler, and a sample is withdrawn within the sampler.

Trier
Consisting of a stainless steel or brass tube that is cut in half (lengthwise) and having a sharpened tip to penetrate the material to be sampled, a trier is used to sample sticky (mud-like) sludge.

Trip Blank
Created by filling a clean sample bottle with deionized water in the field during sampling activities. The sample is handled in the same way as other samples taken from the field. Field blanks are submitted to the lab along with all other samples and are used to detect any contaminants that may be introduced during sample collection, fixing, storage, analysis, and transport.

Turn-Around Time
For an environmental laboratory, the elapsed time between sample receipt and the reporting of analytical results in the form of a data package.

Vectors
Insects, birds, rodents, and domestic animals that are attracted to sewage sludge as a food source and may transport sewage sludge, and pathogens from sewage sludge, to humans.

Vector Attraction Reduction
The technological or management options for treating sewage sludge to the point at which vectors are no longer attracted to sewage sludge or the placement of a barrier between the sewage sludge and the vector.

Appendix L

Regional Regulatory Contact Information

The following information can be used to contact biosolids regulatory staff at U.S. EPA Region 1, as well as the New England states and New York.

This page is intentionally blank.

REGIONAL REGULATORY CONTACT INFORMATION

EPA Region 1

Thelma Murphy
US EPA – New England
1 Congress Street, Suite 1100 (CMU)
Boston, MA 02114-2023
(617) 918-1615
murphy.thelma@epa.gov

Connecticut

Gary Johnson
CT DEP – Water Compliance Unit
79 Elm Street
Hartford, CT 06106-5127
(860) 424-3754
gary.johnson@po.state.ct.us

Maine

Sludge and Residuals Unit Supervisor
ME DEP Sludge Residuals Unit
17 State House Station
Augusta, ME 04333-0017
(207) 287-7826

Massachusetts

Mark Casella
MA DEP
1 Winter Street, 6th Floor
Boston, MA 02108-4747
(617) 654-6517
mark.casella@state.ma.us

New Hampshire

Michael Rainey
NH DES – Residuals Management
P.O. Box 95
6 Hazen Drive
Concord, NH 03302-0095
(603) 271-2818
mrainey@des.state.nh.us

New York

Sally Rowland
NYS DEC – Div. of Solid and Hazardous Materials
625 Broadway
Albany, New York 12233-7253
(518) 402-8704
sjrowlan@gw.dec.state.ny.us

Rhode Island

Alexandre Pinto
RI DEM – Office of Water Resources
235 Promenade Street
Providence, RI 02908-5767
(401) 222-4700
alex.pinto@dem.ri.gov

Vermont

Cathy Jamieson
VT DEC – Residuals Management Section
103 South Main Street, Sewing Building
Waterbury, VT 05671-0405
(802) 241-3831
cathy.jamieson@state.vt.us

This page is intentionally blank.

We Value Your Feedback

Please notify us if you discover mistakes or omissions in this document. Submissions can be sent electronically, mailed, or faxed to:

New England Interstate Water Pollution Control Commission
ATTN: Biosolids Sampling Guide
116 John Street
Lowell, MA 01852-1124
Tel: 978/323-7929
Fax: 978/323-7919
mail@neiwpcc.org

Brief description of error or omission:

_____

_____

_____

_____

Suggested improvement:

_____

_____

_____

_____

General comments:

_____

_____

_____

_____

Can we contact you for additional information? If so please provide contact information:

_____

_____

_____

Thank You.

This page is intentionally blank.