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*