Chapter 6

Sampling and Analysis

The Part 503 rule requires sampling and analysis of biosolids for certain pollutants (metals) and pathogens and for vector attraction reduction if the biosolids are land applied, placed on a surface disposal site, or incinerated. The rule prescribes the frequency for monitoring and lists analytical methods that must be used to analyze different types of samples. The rule does not, however, provide specific instructions on how to sample. This chapter provides general information on sampling biosolids; gives an overview of sampling requirements concerning biosolids incinerator emissions; discusses the methods required by the Part 503 rule for analyzing biosolids samples; and lists publications that provide detailed information about biosolids sampling and analysis.

Guidance for Sampling Biosolids

A number of considerations relate to the care that must be taken in sampling and analyzing biosolids as well as the number of samples that must be taken to be representative. These factors include the size of the sample of biosolids material that is actually being analyzed, the accuracy of the analytical technique, the presence of other materials that might interfere with the analysis, the stability of the analyte being determined, and the potential reduction in volatile solids content of the biosolids when analyzed. Since this chapter provides only general information on sampling and analyzing biosolids, persons responsible for complying with the Part 503 rule should seek additional guidance. Sources of guidance include EPA's ***POTW Sludge Sampling and Analysis Guidance Document***, the EPA

Sewage Sludge Sampling Video, and ***Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge*** (see References), as well as the general Part 503 sampling requirements (summarized in Table 6-1) and the references to specific required analytical protocols listed in Table 6-7. Other guidance documents also are listed at the end of this chapter. If additional information is needed, the reader can contact the Regional EPA permitting authority and, where applicable, the State biosolids contact person.

Who Must Sample?

In most cases, the preparer of biosolids (usually the owner/operator of a treatment works) will be responsible for sampling the biosolids for metals, pathogens, and, where applicable, for vector attraction reduction. Often the generator is also the preparer, land applier, surface disposer, or incinerator of the biosolids. Sometimes a person other than the generator is the preparer (e.g., a person who provides additional processing that may alter the quality of the biosolids before their use or disposal). That preparer may also be required to sample the additionally processed biosolids before they are land applied, surface disposed, or incinerated. Also, the owner/operator of a surface disposal site is responsible for sampling metals under certain circumstances: when needed to meet site-specific limit requirements, or when the boundary of an active biosolids unit is less than 150 meters from the property line of the surface disposal site (see Chapter Three).

How Often Should Sampling Be Done?

The Part 503 rule includes tables listing minimum monitoring frequencies for biosolids that will be land applied, placed on a surface disposal site, or incinerated. Frequency of monitoring requirements range from once a year for facilities using or disposing of relatively small amounts of biosolids to once a month for facilities using or disposing of larger amounts of biosolids. Table 6-2 lists the frequency of monitoring requirements in Part 503. Monitoring must take place at least as often as the table indicates to demonstrate compliance with Part 503 pollutant limits and pathogen and vector attraction reduction requirements.

A number of factors were considered in establishing the frequency of monitoring requirements for the Part 503 rule. The intent was to avoid imposing any undue burden on persons preparing smaller quantities of biosolids. Also, the intent was to require sufficiently frequent monitoring, representative sampling, and quality-assured and -controlled analytical procedures so that the data collected accurately represent the metal content and pathogen and vector attraction reduction status of the biosolids being used or disposed.

TABLE 6-1
Summary of Biosolids Sampling Considerations[a]

Factors To Consider in Developing a Sampling Program	
Who Must Sample?	Preparer, land applier, surface disposer, or incinerator of biosolids.
What Must Be Sampled?	Biosolids: Metals (land application, surface disposal, incineration). Pathogens and vector attraction reduction (land application and surface disposal sites only). Nitrogen (land application only). Biosolids incinerator emissions: Total hydrocarbons (or carbon monoxide), oxygen, temperature, information needed to determine moisture content, and mercury and beryllium, when applicable. Other: Methane gas in air (surface disposal sites only).
How Often Should Sampling Be Done?	From once a year to once a month, depending on the amount of biosolids used or disposed (see Table 6-2).
How Should Sampling Be Done and How Many Samples Should Be Taken?	Take either: Grab samples[b] (individual samples) for pathogens and percent volatile solids determinations, or Composite samples[b] (several grab samples combined) for metals. No fixed number of individual samples required (except for Class B pathogens, Alternative 1, take 7 samples). Enough material must be taken for the sample to be representative. Take a greater number of samples if there is a large amount of biosolids or if characteristics of biosolids vary a lot. See Table 6-4 for guidance (e.g., continuous, instantaneous, or monthly averages required).
When To Sample?	Before use or disposal. If biosolids are used or disposed before sampling results are available, and the results subsequently show that a regulatory limit is exceeded the responsible person will be in noncompliance with Part 503. See also Table 6-3.
Where To Collect Samples?	Usually at site of preparer (e.g., treatment works). Sometimes samples must be collected at land application or surface disposal sites. Sample from moving biosolids when possible to obtain a well-mixed sample. If you must sample from a stationary location, the sample should represent the entire area. Appropriate sampling points differ for liquid or dewatered biosolids (see Table 6-5).
What Size of Sample, Sample Equipment, Storage Times?	See Table 6-6.
What Methods Should Be Used To Analyze Samples?	Part 503 requires that specific analytical methods be used for different types of samples (see Table 6-7).

[a] All information in this table is discussed in more detail in the text of this chapter.

[b] Guidance, not a Part 503 rule requirement.

TABLE 6-2
Frequency of Monitoring for Land Application, Surface Disposal, and Incineration of Biosolids

Amounts of Biosolids* (metric tons per 365-day period)	Frequency
Greater than zero but less than 290	Once per year
Equal to or greater than 290 but less than 1,500	Once per quarter (four times per year)
Equal to or greater than 1,500 but less than 15,000	Once per 60 days (six times per year)
Equal to or greater than 15,000	Once per month (twelve times per year)

* Amount of biosolids (other than domestic septage) land applied, placed on an active biosolids unit, or fired in an incinerator—dry-weight basis.

Monitoring frequency should anticipate the potential for changes in metals concentration, pathogen density, and vector attractiveness in biosolids. In general, metals contents will change little unless there is a significant reduction in the volatile solids content of the biosolids. In contrast, bacterial pathogens (not enteric viruses and viable helminth ova) can regrow in biosolids under certain conditions. Moreover, the extent of vector attraction reduction achieved using Alternatives 6 (pH adjustment) or Alternatives 7 or 8 (drying) may change.

Monitoring frequency also should take into account when biosolids are actually being used or disposed. The rule assumes, especially in regard to preparers of large amounts of biosolids, that the biosolids will be used or disposed consistently throughout the year. If biosolids are being stored for a number of months before use or disposal, a large mass could accrue. Although the Part 503 rule does not require analysis until the biosolids are used or disposed, the preparer, land applier, or disposer might want to take composite samples for analysis throughout the storage period so that sampling results are more representative and the operation affords better process control. Remember, however, that the fecal coliform and *Salmonella* sp. determinations (for Class A and B pathogen alternatives, where applicable) have to be made sufficiently close to the time biosolids are actually used or disposed to be indicative of whether the potential for regrowth has been controlled.

In general, if a person is operating in such a manner that the biosolids being used or disposed meet applicable Part 503 requirements, continuing in this manner of operation would tend to minimize the likelihood of subsequent noncompliance. Thus, a monitoring program that enables one to ensure that critical operating parameters continue to be met is good practice.

Given the varying nature of the many different processes that need to be monitored for and controlled, it is not possible to provide one simple guidance suggestion for when to monitor for various Part 503 required parameters. Table 6-3 summarizes additional important monitoring considerations for each of the various parameters.

How Many Samples Should Be Taken?

Although the Part 503 rule establishes frequency of monitoring requirements for biosolids, it does not specify how many samples need to be taken. (There is one exception—for Class B, Alternative 1 pathogen requirements, the regulation states that seven samples must be collected.) Is one sample enough for most monitoring? Are 20 too many? The appropriate number of samples to take depends on conditions at each site. More than one sample is usually necessary to accurately represent a particular stream or batch of biosolids. ***The key is to obtain a representative sample,*** as is required by the Part 503 rule.

In general, the more samples taken, the greater the chance that the sampling results will be representative of the biosolids at a particular facility. Also, the larger the amount of biosolids a facility uses or disposes, the greater the number of samples that will be needed to obtain a representative sample. A greater number of samples should be taken if the characteristics of the biosolids vary considerably (e.g., if the solids content or pathogen levels vary significantly from one batch to another). Table 6-4 provides guidance on the types of biosolids samples that must be collected to assess the level of metals and pathogens, and to monitor other parameters. The type of parameter limit (e.g., instantaneous, monthly averages) will affect the determination of how many samples should be taken.

Important factors to consider when determining how many samples to take include:

Standard deviation. Find out the extent of the variation from the average result (the mean). This concept is known as the standard deviation. The standard deviation is determined by taking the square root of the arithmetic average of the squares of the deviations from the mean in a frequency distribution. The greater the standard deviation, the greater the number of individual samples that should be taken to get a representative sample.

Addition of commercial/industrial pollutants to sewage system. Determine whether pollutants are being added (or "cycled") into the sewage system by commercial or industrial processes. If cycling of pollutants is occurring, it is advisable to collect more samples to ensure that they include the high pollutant levels (or "spikes") that can come from commercial or industrial discharges.

TABLE 6-3
Monitoring Considerations for Key Parameters

Parameter	Validity of Analytical Data over Time and When Sampling/Analysis Must Occur
	METALS
Metals	Data remain valid for biosolids if no significant change in volatile solids. Determine monitoring frequency in accordance with monitoring frequency requirements.
	PATHOGENS CLASS A
Applies to All Class A Pathogen Reduction Alternatives (PRA):	Because regrowth of fecal coliform and *Salmonella* sp. can occur, monitoring should be done sufficiently close to the time of biosolids use or disposal so data are available and no additional regrowth occurs: (a) before land application or suface disposal, or (b) when biosolids are prepared for sale or give-away in a bag or other container for land application, or (c) when biosolids are prepared to meet EQ requirements. Once destroyed, enteric viruses and viable helminth ova do not regain viability.
	Additional Information on Each Class A Pathogen Category
Class A PRA 1: Thermal Treatment, Moisture, Particle Size & Time Dependent	Data remain valid as long as biosolids remain dry before use. Time, temperature, and moisture content should be monitored continuously to ensure effectiveness of treatment.
Class A PRA 2: High pH, High Temperature	Monitor to ensure that pH 12 (at 25°C) is maintained for more than 72 hours.
Class A PRA 3: Enteric Virus & Viable Helminth Ova To Establish Process	To establish a process, determine with each monitoring episode until the process is shown to consistently achieve this status. Then monitor process at sufficient frequency to ensure its validity.
Class A PRA 4: Enteric Virus & Viable Helminth Ova for Unknown Process	Do not know whether enteric virus or viable helminth ova were present and destroyed or just not detected. Monitor representative sample of biosolids material: (a) to be used or disposed, or (b) when prepared for sale or give-away in a bag or other container for land application, or (c) when prepared to meet EQ requirements.
Class A PRA 5: PFRP	Monitor at sufficient frequency to show compliance with time and temperature or irradiation requirements in Table 5-6.
Class A PRA 6: PFRP Equivalent	Monitor at sufficient frequency to show compliance with PFRP or equivalent process requirements.

TABLE 6-3 (continued)
Monitoring Considerations for Key Parameters

	PATHOGENS CLASS B
Class B PRA 1: Fecal Coliform	Measure the geometric mean of 7 samples taken over a 2-week period sufficiently close to the time of use or disposal so that (i) data are available and (ii) no additional regrowth occurs before use or disposal.
Class B PRA 2:	Monitor at sufficient frequency to show that the PSRP requirements in Table 5-7 are met.
Class B PRA 3:	Monitor at sufficient frequency to show that the equivalent PSRP requirements are met.
	VECTOR ATTRACTION REDUCTION
Vector Attraction Reduction (VAR) 1: 38% Volatile Solids Reduction (VSR)	Once achieved, no further attractiveness to vectors. If a batch process, determine VSR for each batch. If for a continuous process, determine VSR based on material being put in and withdrawn. Monitor at sufficient frequency to verify that the necessary VSR operating conditions are met.
VAR 2 for Anaerobic Digestion: If Cannot Meet VAR 1 Lab Test **VAR 3 for Aerobic Digestion: If Cannot Meet VAR 1 Lab Test** **VAR 4: SOUR Test for Aerobic Processes**	Once achieved, no further attractiveness to vectors. If a batch process, determine VSR for each batch. If unable to show VSR, then conduct lab test. Monitor at sufficient frequency to verify that biosolids are meeting the necessary operating conditions.
VAR 5: Aerobic >40°C	Monitor at sufficient frequency to show that biosolids are achieving an average temperature of 45°C over a 2-week period.
VAR 6: Adding Alkaline Material	Determine pH over time for each batch. VAR has been achieved as long as the pH does not drop such that putrefaction begins prior to land application or surface disposal.
VAR 7: Moisture Reduction No Unstabilized Primary Solids	To be achieved only by the removal of water. VAR has been achieved as long as the moisture level remains below 30%.
VAR 8: Moisture Reduction Primary Unstabilized Solids	To be achieved only by the removal of water. VAR has been achieved as long as the moisture level remains below 10%.

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TABLE 6-3 (continued)
Monitoring Considerations for Key Parameters

VAR 9: Injection into Soil	No significant amount of biosolids remains on soil surface within 1 hour after injection.
VAR 10: Incorporation into Soil	Biosolids must be incorporated into soil within 6 hours after being placed on the soil surface.
VAR 11: Covered with Soil Surface Disposal	Surface disposed biosolids must be covered daily.
VAR 12: Domestic Septage pH Adjustment	Preparer must ensure that pH is 12 for more than 30 minutes for each batch of domestic septage treated with alkaline material.

Results of previous samples. If previous sampling results show that biosolids contain pollutants or pathogens at levels close to the regulatory limits specified in Part 503, then consider taking a greater number of samples to determine if the biosolids are approaching or have reached the regulatory limit. The closer the biosolids come to the regulatory limits, the more critical sampling results become.

Whether the biosolids are well mixed. Well-mixed biosolids provide a more representative sample. If a particular batch or stream of biosolids is well mixed, then fewer samples need to be taken. If the biosolids are not well mixed, then more samples should be taken.

Special methods have been developed by EPA's Office of Wastewater Management to determine how many samples should be collected when biosolids must be sampled at land application or surface disposal sites rather than where generated. These methods involve the use of mathematical concepts such as sample means, standard deviations, and confidence intervals, which are explained in EPA's ***POTW Sludge Sampling and Analysis Guidance Document*** (see References).

How Is Sampling Done?

There are two basic types of samples: grab samples and composite samples. Because a grab sample is a single sample collected at a specific time and location, it is representative of the composition of a material being sampled only at that particular moment and place.

The other type of sample, the composite sample, is made up of several grab samples taken over a period of time and/or from different locations. In most cases, a composite sample is more representative than a grab sample because the composite can reveal information about the composite's subsamples of material from several locations and time periods. Thus,

TABLE 6-4
Types of Limits for Which Sampling Must Be Done

Use or Disposal Practice	Parameter	Nature of Determination
Land Application	Pollutant Limits:	
	Ceiling Limit Concentrations (Table 2-1 in this document, or Table 1 in Part 503.13)	Instantaneous—may not be exceeded
	Pollutant Concentrations—PC or EQ biosolids (Table 2-1 in this document, or Table 3 in Section 503.13)	Monthly averages
	Nitrogen	Representative value used to determine agronomic rate
	CPLR (Table 2-1 in this document, or Table 2 in Section 503.13)	May not be exceeded at any site
	APLR (Table 2-1 in this document, or Table 4 in Section 503.13)	May not be exceeded during a 365-day period
Surface Disposal	Methane gas	Continuously monitored in air; instantaneous—may not be exceeded
	Metals	Instantaneous—may not be exceeded
Incineration	Metals (except beryllium and mercury)	Daily concentration; if required to report once per month, average of each day operated during the month
	Total hydrocarbons (THC) or Carbon-monoxide (CO)	Continuously monitored; monthly average is reported, which is the arithmetic mean of hourly averages with a minimum of 2 readings per hour
	Oxygen	Continuously monitored
	Temperature	Continuously monitored
	Moisture	Continuously monitored

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TABLE 6-4 (continued)
Types of Limits for Which Sampling Must Be Done

Use or Disposal Practice	Parameter	Nature of Determination
Land Application and Surface Disposal	Pathogens (or Indicators):	
	Class A Pathogens: Fecal coliform	Part 503 rule specifies a density of <1,000 fecal coliform/g total solids (dry-weight basis). Guidance (EPA/625/R-92/013) suggests the geometric mean of a minimum of 7 individual grab samples taken over a 14-day period, similar to the fecal coliform determination for Class B.
	Salmonella sp.	Part 503 rule specifies a density of <3 MPN *Salmonella* sp./4 g total solids (dry-weight basis). Guidance (EPA/625/R-92/013) suggests the arithmetic mean of a minimum of 7 individual grab samples taken over a 14-day period.
	Enteric virus	Part 503 rule specifies a density of <1 PFU/4 g total solids (dry-weight basis). Guidance (EPA/625/R-92/013) suggests that one composite sample of 7 grab samples be made over a 14-day period and that the arithmetic mean of 4 duplicate analyses of that composite be determined.
	Viable helminth ova	Part 503 rule specifies a density of <1 viable ova/4 g total solids (dry-weight basis). Guidance (EPA/625/R-92/013) suggests that one composite sample of 7 grab samples be made over a 14-day period and that the arithmetic mean of 4 duplicate analyses of that composite be determined.
	Class B Pathogens: Fecal coliform	Part 503 rule specifies the geometric mean of 7 individual samples. Guidance suggests that they be taken over a 14-day period; the rule states that the geometric mean may not exceed ≤2 million MPN or CFUs/g total solids (dry-weight basis).

Note: CFUs = colony-forming units
MPN = most probable number

whenever possible and appropriate, composite sampling should be conducted (e.g., for metals). Take several grab samples, combine them, and then send the composite sample to a laboratory for analysis.

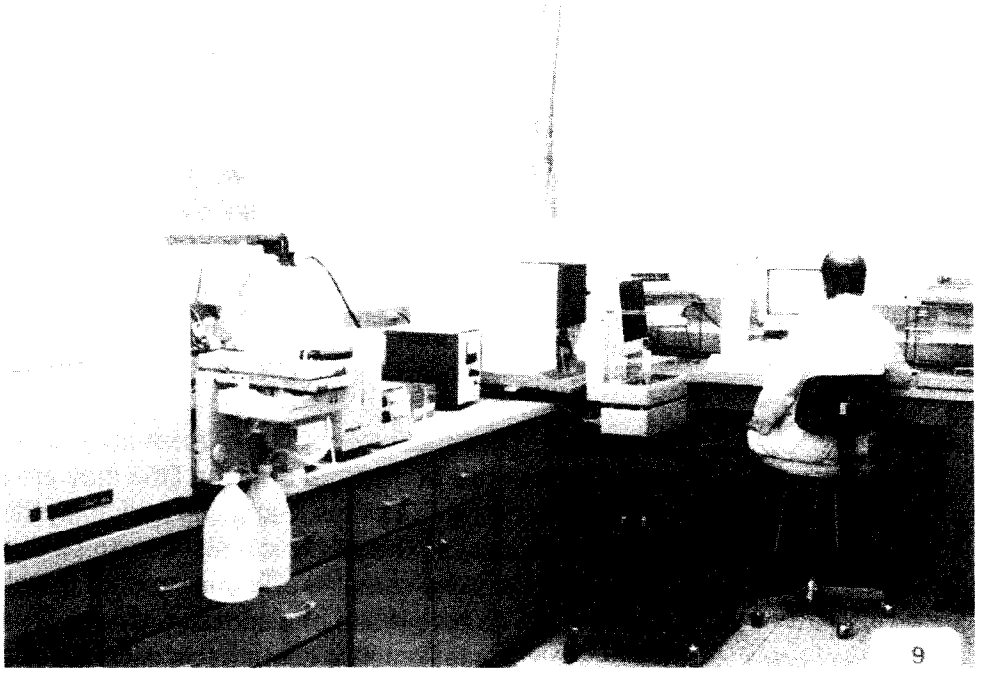
Although composite samples taken over one to several weeks and properly stored are generally more representative than composite samples gathered over a short timeframe, certain tests require a composite sample that has been gathered over a short period of time. This is because tests of biosolids for certain analytes, such as pathogens, can become invalid due to ease of contamination, regrowth, or rapid die-off.

When Should Samples Be Taken?

Part 503 states that biosolids must meet the requirements of the rule at the time of their use or disposal or at the time they are prepared if distributed in bags or meeting EQ status. Sampling and analysis should take place before use or disposal so that analytical results can be available ahead of time. Biosolids could be sampled and analyzed for metals content a considerable period of time before use or disposal, provided no significant additional reduction in volatile solids content has occurred. Certain pathogen and vector attraction reduction determinations, however, would need to be made close to the time of use or disposal to meet the rule's requirements. In some cases (e.g., with some of the pathogen and vector attraction reduction alternatives) sampling may need to be conducted over the applicable period of time to show that reduction of parameters has been achieved. See also Tables 6-3 and 6-4.

Waiting to establish sampling results before use or disposal is critical to avoid exceeding limits if the levels of one or more pollutants or pathogens in the biosolids being tested are close to the regulatory limits or if there is a high potential for pollutant spikes. If initial sampling results for a particular biosolids material indicate that pollutant levels are well below the regulatory limits, later sampling results might also be expected to show that pollutant levels will not exceed those limits. If, however, you suspect that pollutant levels are close to the regulatory limits, then waiting for results before using or disposing of the biosolids will avoid a situation in which a detected exceedance results in noncompliance.

Establishing compliance before use or disposal also helps ensure that a particular batch of biosolids is available for additional sampling if necessary. For example, suppose that a batch of biosolids was land applied before sampling results were returned from the laboratory and that when the sampling results became available, they indicated unusually high levels of a pollutant in excess of the regulatory limit. Resampling might be appropriate to determine whether a laboratory error was made. If biosolids have already been used or disposed prior to an exceedance determination, the permitting authority would have to decide what actions to take to ensure protection of



A laboratory technician analyzes samples of composted biosolids in Aurora, Illinois.

public health and the environment. The enforcement authority also would have to make a determination about actions it might take that could lead to penalties and fines.

Where Should the Samples Be Taken?

In general, more representative sampling occurs when the biosolids being sampled are moving rather than stationary. The movement of biosolids tends to cause mixing and thus a more uniform entrainment of solids and pollutants. Depending on the type of biosolids material (liquid, dewatered, or dried) and the treatment process, certain sampling points will provide better samples. Table 6-5 lists some of the better places to sample biosolids.

Liquid biosolids should generally be sampled from pipelines, or preflushed pipeline ports. Whenever possible, the sampling locations should be as far downstream in the treatment works as possible to take advantage of the maximum mixing that will occur and to capture the most representative sample of biosolids that will be used or disposed. For example, sampling before digestion would not be representative of the pathogen or metals levels that would be present after digestion. Sometimes liquid biosolids may need to be sampled from lagoons. This should be done in such a way that the floating, suspended, and sediment layers of the biosolids are all included. The sample can be obtained using a liquid waste sampler, known as a coliwasa (described in EPA Solid Waste Method 846), or, if the biosolids are quite thick, by using a coring device.

TABLE 6-5
Sampling Points for Biosolids

Biosolids Type	Sampling Point
Anaerobically Digested	Collect sample from taps on the discharge side of positive displacement pumps.
Aerobically Digested	Collect sample from taps on discharge lines from pumps. If batch digestion is used, collect sample directly from the digester. Cautions: 1. If biosolids are aerated during sampling, air entrains in the sample. Volatile organiccompounds may be purged with escaping air. 2. When aeration is shut off, solids may settle rapidly.
Thickened	Collect sample from taps on the discharge side of positive displacement pumps.
Heat Treated	Collect sample from taps on the discharge side of positive displacement pumps *after* decanting. Be careful when sampling heat-treated biosolids because of: 1. High tendency for solids separation. 2. High temperature of sample (temperature >60°C as sampled) can cause problems with certain sample containers due to cooling and subsequent contraction of entrained gases.
Dewatered, Dried, Composted, or Thermally Reduced	Collect sample from material collection conveyors and bulk containers. Collect sample from many locations within the biosolids mass and at various depths.
Dewatered by Belt Filter Press, Centrifuge, Vacuum Filter Press	Collect sample from biosolids discharge chute.
Dewatered by Biosolids Press (plate and frame)	Collect sample from the storage bin; select four points within the storage bin, collect equal amount of sample from each point and combine.
Dewatered by Drying Beds	Divide bed into quarters, grab equal amounts of sample from the center of each quarter and combine to form a composite sample of the total bed. Each composite sample should include the entire depth of the biosolids material (down to the sand).
Compost Piles	Collect sample directly from front-end loader while biosolids are being transported or stockpiled within a few days of use.

A more representative sample of dewatered biosolids (e.g., with a solids content of 10 to 40 percent) can be obtained by sampling from moving conveyor belts or front-end loaders that are moving a pile of biosolids (i.e., biosolids from drying beds, outdoor drying windrows, compost storage piles, or dried-out lagoons should be sampled, if possible, when moved). If the biosolids sample must be taken with the biosolids in place, samples from the entire area should be taken and combined (e.g., samples from a compost pile should be taken at various depths and along the length of the pile and then mixed together).

In most cases, biosolids are sampled at the end of a treatment process, just prior to their use or disposal. In some instances, sampling may need to be carried out at a storage, surface disposal, or land application site because of the possibility of a change in pollutant, pathogen levels, or vector attractiveness during the period between treatment and use or disposal.

What Types of Sampling Equipment Should Be Used?

Sampling equipment (e.g., coring devices, coliwasas, pitchers, conduits, shovels, trowels, containers) must be made of materials that will not contaminate or react with the biosolids. Suitable sampling equipment materials generally include glass, stainless steel, and plastic (Teflon, polyethylene, polypropylene). Any steel equipment used must not be galvanized or zinc coated because it will contaminate the sample. Moreover, all equipment should be kept clean to avoid contamination. For samples used to demonstrate compliance with Class A pathogen requirements, sampling equipment should be sterilized prior to sampling. Requirements for sample containers are often listed in the description of the analytical method (see below).

How Large a Sample Is Needed? How Long Can the Sample Be Stored?

It is important both to collect the correct amount of biosolids needed to perform sample analysis and to preserve and store samples properly. Table 6-6 lists appropriate containers, sample sizes, and preservation and storage times for sampling biosolids for metals and pathogens. Wide-mouthed containers are recommended for biosolids sampling.

What If a Test Result Does Not Meet the Part 503 Requirements?

To answer this question, it is important to clarify, first of all, that no violation occurs unless biosolids have been used or disposed and pollutant contents exceed regulatory requirements. Second, it is necessary to clarify whether a monthly average determination (e.g., for pollutant concentration limits or Class B pathogen status for land application) or an instantaneous determination (e.g., ceiling concentration limit for land application) is at issue. Land application would be in compliance even if some of the daily or weekly biosolids metal determinations included in the monthly average exceeded the pollutant concentration regulatory limits but the averages did not. Likewise, land application would be in compliance, even if one or more of the 7 fecal coliform densities exceeded the Part 503 regulatory limit, provided the geometric mean of all 7 densities did not.

Consider the same question for land application ceiling concentration limits. What if one of several samples of the mass of biosolids being analyzed was above the Part 503 regulatory limit? Does this mean that the particular batch of biosolids is out of compliance and cannot be land applied? The answer is

TABLE 6-6
Proper Conditions for Biosolids Sampling

Parameter	Wide-Mouthed Container	Preservative[a]	Maximum Storage Time[a]	Minimum Volume[b]
Metals				
Solid and semi-solid samples	P,G	Cool, 4°C	6 months	300 mL
Liquid (mercury only)	P,G	HNO_3 to pH <2	28 days	500 mL
Liquid (all other liquid metals)	P,G	HNO_3 to pH <2	6 months	1,000 mL
Pathogen Density and Vector Attraction Reduction				
Pathogens	G,P,B,SS	1. Cool in ice and water to <10°C if analysis delayed >1 hr, or	6 hours	1-4 liters[c]
		2. Cool promptly to <4°C, or	24 hours (bacteria and viruses) 1 month (helminth ova)	
		3. Freeze and store samples to be analyzed for viruses at 0°C[d]	2 weeks	
Vector attraction reduction		Varies[b]	Varies[b]	1-4 liters[c]

[a] Preservatives should be added to sampling containers prior to actual sampling episodes. Storage times commence upon addition of sample to sampling container. Shipping of preserved samples to the laboratory may be, but is generally not, regulated under Department of Transportation hazardous materials regulations.

[b] Varies with analytical method. Consult 40 CFR Parts 136 and 503.

[c] Reduced at the laboratory to approx. 300 mL samples.

[d] Do not freeze bacterial or helminth ova samples.

P = Plastic (polyethylene, polypropylene, Teflon)

G = Glass (non-etched Pyrex)

B = Presterilized bags (for dewatered or free-flowing biosolids)

SS = Stainless steel (not steel- or zinc-coated)

Source: 40 CFR Part 136, and EPA, December 1992.

yes, the material could not be land applied unless treated to reduce the ceiling concentration below the regulatory limit.

If a sample result were discovered to exceed certain limits after the biosolids are land applied for any of the above cases, the biosolids would be out of compliance and the responsible person would be subject to enforcement actions. The permitting authority will decide what action to take when a requirement is not met. If the failure is substantial, the permitting authority might withhold approval for—or may no longer allow—land application, surface disposal, or incineration of the biosolids. If the failure is slight, the permitting authority might allow reasonable efforts to be made to bring the process into compliance.

Determining the accuracy of a given sample result affects the ability to achieve compliance. In many sampling efforts, some results might not accurately represent the material being sampled as well as other samples would (these are known as outliers). Some outliers may indicate noncompliance when other, more representative samples show compliance. Also, laboratory errors may indicate sample failure when in fact the sample should have passed. To account for outliers and lab errors, the person doing the monitoring should take a greater number of samples over a long timeframe. If most samples in a rigorous sampling effort show compliance, it is more likely that a single sample failure is an outlier or due to laboratory error. It should be noted, however, that a large number of samples is needed to prove that a sample result is an outlier.

Other Sampling Considerations

Other factors that need to be considered when developing sampling procedures for biosolids include:

Regrowth potential for bacteria. Under certain conditions, some types of bacteria may regrow in biosolids. The possibility for regrowth depends on whether conditions such as temperature, pH, and other factors make the biosolids an advantageous food source for the bacteria. Because of this potential, the Part 503 rule requires sampling for fecal coliform or *Salmonella* sp. bacteria as close as possible to the time of biosolids use or disposal for the Class A and B pathogen alternatives where such determinations are required.

Proper quality assurance and quality control procedures. QA/QC procedures appropriate for collecting samples for metals and for performing microbiological analysis should be defined and followed. (EPA's ***POTW Sludge Sampling and Analysis Guidance Document*** provides guidance on sampling and QA/QC procedures. See also, lists of contacts at the end of this document.)

Packaging procedures. Ensure that packaging does not alter the biosolids' character or quality. Shipping containers should be kept upright, tightly sealed, cushioned, insulated, and refrigerated to keep the sample at approximately 4°C but without freezing the sample.

Shipping time. The sample should reach the lab within 24 hours to ensure that proper temperature conditions are maintained. U.S. Department of Transportation regulations prohibit shipping acidified samples without the proper manifest and hazard markings. Use of acidified samples should be avoided because shipping is costly.

Proper sample documentation. Documentation is important for ensuring QA/QC. Documentation includes clearly marked labels with all appropriate identifying information; a chain-of-custody record that documents the transfer of the sample material from person-to-person; and a log book that records all sampling activities.

Personnel safety. Personnel handling biosolids samples should take precautions to minimize contact with pathogens and pollutants that may be present in biosolids. Rubber or latex gloves and waterproof garments should be worn to prevent direct contact. Personnel should follow procedures that limit the production of explosive gases within the samples; preserving and refrigerating samples suppresses biological activity that produces such gases. In addition, sampling personnel should take precautions to avoid injury when sampling high-pressure biosolids lines or lines containing high-temperature, thermally conditioned biosolids.

Sampling Emissions for Biosolids Incineration

Another type of sampling required by Part 503 concerns metals in biosolids fired in an incinerator and total hydrocarbon (THC) (or carbon monoxide—CO) emissions from the incinerator's exhaust stack. Methods for sampling biosolids incinerator emissions are very detailed and should be performed by experienced professionals.

Chapter Four provides a brief discussion of the performance testing required for biosolids incinerators. Testing personnel will sample biosolids, sample the stack gases, and document the operating conditions of the furnace during the test (i.e., temperature, pressure, voltage, and operation of air pollution control devices). The performance test, which is run to represent normal incinerator operating conditions, generates information that is used to establish acceptable operating conditions for the incinerator and to calculate maximum pollutant levels for the biosolids fired in the incinerator.

Maintaining compliance with the required THC (or CO) regulatory limits is also discussed in Chapter Four of this document.

Analytical Methods for Biosolids Samples

The Part 503 rule requires that specific methods be used for analyzing biosolids samples for metals, pathogens, and vector attraction reduction. Table 6-7 lists these methods.

Sources of Additional Information on Sampling and Analysis of Biosolids

Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge
U.S. EPA, Office of Research and Development, Cincinnati, OH. Phone (614) 292-6717. EPA/625/R-92/013, December 1992.

POTW Sludge Sampling and Analysis Guidance Document
U.S. EPA, Permits Division, Washington, DC. 1st edition, August 1989. NTIS PB 93-227957. Phone (800) 553-6847. Revised 2nd edition scheduled for publication in September 1994.

TABLE 6-7
Analytical Methods for Biosolids Sampling[a]

Sample Type	Method
Enteric Viruses	ASTM Designation: D 4994-89, Standard Practice for Recovery of Viruses from Wastewater Sludges, Annual Book of ASTM Standards: Section 11. Water and Environmental Technology, ASTM, Philadelphia, PA, 1992.
Fecal Coliform	Part 9221 E or Part 922 D, Standard Methods for the Examination of Water and Wastewater, 18th edition, American Public Health Association, Washington, DC, 1992.
Helminth Ova	Yanko, W.A., Occurrence of Pathogens in Distribution and Marketing Municipal Sludges, EPA/600/1-87/014, 1987. PB 88-154273/AS, National Technical Information Service, Springfield, VA; (800) 553-6847.
Inorganic Pollutants	Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW-846, 3rd edition (1986) with Revision I. 2nd edition. PB 87-120291, National Technical Information Service, Springfield, VA. 3rd edition Doc. No. 955-001-00000-1, Superintendent of Documents, Government Printing Office, Washington, DC.
***Salmonella* sp. Bacteria**	Part 9260 D, Standard Methods for Examination of Water and Wastewater, 18th edition, American Public Health Association, Washington, DC, 1992; or, Kenner, B.A. and H.P. Clark, Detection and Enumeration of *Salmonella* and *Pseudomonas aeruginosa*, J. Water Pollution Control Federation, 46(9):2163-2171, 1974.
Specific Oxygen Uptake Rate	Part 2710 B, Standard Methods for the Examination of Water and Wastewater, 18th edition, American Public Health Association, Washington, DC, 1992.
Total, Fixed, and Volatile Solids	Part 2540 G, Standard Methods for the Examination of Water and Wastewater, 18th edition, American Public Health Association, Washington, DC, 1992.
Percent Volatile Solids Reduction Calculation[b]	Environmental Regulations and Technology—Control of Pathogens and Vectors in Sewage Sludge, EPA/625/R-92/013, U.S. Environmental Protection Agency, Cincinnati, OH, 1992; (614) 292-6717.

[a] These analytical methods are required by the Part 503 rule.

[b] This analytical method is provided as guidance in the Part 503 rule.

Common Questions and Answers

Q: Most facilities will not wait several weeks for laboratory results before shipping biosolids off site for use or disposal. What action will be taken when the laboratory results show that biosolids that have already been land applied actually exceeded pollutant ceiling concentration limits?

A: The preparer will be in violation for noncompliance. The ceiling concentration limits in Part 503 are instantaneous, not-to-exceed values; thus, exceeding the concentrations is a violation. If the biosolids quality is unusually close to the ceiling concentration limits, the facility may need to require pretreatment or to take other suitable steps to enhance the quality. EPA will enforce the rule as is warranted, using all remedies at its disposal, including injunctive relief and/or penalty actions.

Q: Are there any measures in the Part 503 rule that deal with small communities that cannot afford to achieve immediate compliance for monitoring biosolids and are not able to receive financial aid?

A: There are no specific provisions in the Part 503 rule that deal with the financial status of a preparer. In developing the frequency of monitoring requirements, however, EPA based the stipulations on the amount of biosolids being used or disposed. Therefore, smaller facilities that use or dispose of smaller amounts of biosolids will generally be required to monitor less frequently than larger facilities.

Q: Do Part 503 frequency of monitoring requirements apply in the same manner to biosolids that are being stored for a significant period of time prior to land application as to those biosolids that are being land-applied throughout the year generally in accordance with their generation or preparation?

A: The frequency of monitoring requirements in the Part 503 rule were established for biosolids that are being land-applied generally in accordance with their generation or preparation. The overriding consideration for monitoring biosolids that have been stored for a period of months prior to being land-applied over a short time period is to obtain a representative sample that reflects the current status of the biosolids.

Q: How should biosolids be monitored that have been stored for a number of months prior to being land applied during a short time period compared to biosolids that are being land applied generally in accordance with their generation or preparation? How frequently should you monitor? What is the potential for liability if the biosolids being land-applied fail to meet the ceiling limits, pollutant concentration limits, or pathogen and vector attraction reduction requirements?

A: The following two cases are given to help answer the frequency of monitoring questions. In both of the following cases the assumption is made that the facility generates or prepares over 15,000 DMT of biosolids during the year. In ***Case 1*** the biosolids are land applied 12 months during the year generally in accordance with their generation/preparation, and in ***Case 2*** they are stored for 11 months and then land applied during the 12th month. The answer for ***Case 1*** is as described in the frequency of monitoring tables in the Part 503 rule. The answer to ***Case 2*** involves the need for taking a sample that is truly representative of the mass of biosolids that is about to be land applied. The exact method chosen to gain a representative sample for ***Case 2*** will depend on a careful examination of the circumstances.

Pollutant Ceiling Limits

For ***Case 1*** the answer is rather straightforward. The frequency of monitoring table in the Part 503 rule requires facilities that are generating or preparing and land applying 15,000 DMT of biosolids throughout the year to take one representative sample for analysis each month. If any analyzed sample exceeded the ceiling limits in a given month, the biosolids represented by that sample could not be land applied unless that batch of biosolids underwent further treatment to reduce the content of the exceedant pollutant to below its ceiling limits. If the exceedant batch of biosolids was added to other non-exceedant batches, then the entire combined batch of biosolids would have to undergo treatment and additional sampling and analysis to show its compliance with pollutant ceiling limits.

For ***Case 2***, the answer is different. The generator, preparer, or land applier, in conjunction with the permitting authority, could decide on more than one approach for frequency of monitoring—as long as sample(s) taken were representative. One approach (Case 2A) might be to take one or more representative composite samples of the entire 15,000 DMT batch of stored biosolids for analysis. Another approach (Case 2B) might be to take 12 monthly representative samples as the biosolids are being generated or prepared and put into storage. Other approaches might also be appropriate.

Failure to meet pollutant ceiling limits for Case 2A monitoring would require additional treatment and testing to show compliance. *If you were following the monthly sampling and analysis Case 2B option, what would happen if one of your monthly samples exceeded the ceiling limits?* The batch of biosolids that the sample represented could not be land applied unless you provided additional treatment to reduce the pollutant levels in the exceedant batch to below the ceiling limits. If the exceedant batch was added to other non-exceedant biosolids of batches, then the entire mixture of batches would have to undergo additional treatment, sampling, and analysis to show that the pollutant ceiling limits were not exceeded.

Additional treatment processes that could be used to reduce the pollutant content in the exceedant biosolids to below the ceiling limits for either ***Case 1 or 2*** could involve composting, lime treatment, or blending with biosolids that contain lower levels of pollutants.

Pollutant Concentration Limits

Assume in ***Case 1*** that you were trying to meet the pollutant concentration limits so that the biosolids could be used with a PC or EQ classification. Assume also that weekly composite samples were taken and analyzed as a basis for determining the average monthly pollutant concentrations.

Could the PC or EQ classification for your biosolids be retained if the pollutant levels in any of the weekly composite samples exceeded the pollutant concentration limits? As long as the average of all of those samples taken within a given month did not exceed the pollutant concentration limit, your biosolids would still have a PC or EQ classification—provided they also met necessary pathogen and vector attraction reduction requirements. (Remember that you must report the results of all analyses made.)

Assume in ***Case 2*** that you were trying to meet the pollutant concentration limits and use the biosolids with a PC or EQ classification. As for Case 2 pollutant ceiling limits, you, in conjunction with the permitting authority, could decide on more than one approach for taking a representative sample of the stored biosolids. For Case 2A suppose that your sampling and analysis was performed just prior to the month the biosolids were being land applied, and that you took more than one representative sample during that month. Suppose further that one of the representative samples exceeded one of the pollutant concentration limits. *Would your 15,000 DMT of biosolids meet the pollutant concentration limits as long as the average of all those representative samples taken during that month meet the limits?* Yes. Depending on the circumstances, however, you might be required to remix the 15,000 DMT of biosolids and take and test several more representative samples to demonstrate compliance with the pollutant concentration limits.

For Case 2B, suppose that all biosolids generated by the facility were commingled during the 11-month storage period and that at least one of the monthly samples exceeded the pollutant concentration limits. Assume further that all the stored biosolids were land applied during the 12th month. *Could the biosolids be classified as PC or EQ if the mean of the 12 monthly analyzed samples did not exceed the pollutant concentration limits?* Yes. Depending on the circumstances, however, you might be required to remix the 15,000 DMT of biosolids and take and test several more representative samples to demonstrate compliance with the pollutant concentration limits.

Cumulative Pollutant Loading Rate (CPLR) Monitoring

Assume that you analyzed for pollutant concentrations as described immediately above and found that the concentration of metal pollutants in your tested biosolids exceeded those concentrations listed in Section 503.13, Table 3, but were less than the ceiling limits in Section 503.13, Table 1. By whatever monitoring method you used to sample representatively and determine pollutant contents, you would have to keep track of and meet all other Part 503 requirements for land applying those biosolids.

Pathogen and Vector Attraction Reduction

For both ***Cases 1 and 2***, the current guidance for determining if biosolids meet Class B pathogen requirements (where pathogen indicator measurements are applicable) is to take a minimum of seven samples for analysis that are representative of the material that will be used or disposed. As long as the geometric mean of the pathogen indicator concentration does not exceed the limit, the biosolids meet the Class B pathogen classification. Guidance also suggests that the same seven samples could be used to determine vector attraction reduction (VAR) for those VAR processes that require analysis. Similar steps could be taken to determine biosolids compliance with Class A pathogen status.

It is important that the samples of the biosolids be taken and analyzed for pathogen and VAR close enough to the time of land application to be reflective of current status, but not so close that the results are not available until after the biosolids have been land applied. If the delayed results showed that the land applied biosolids were not in compliance, then you would be subject to a potential enforcement action.