Ohio EPA

State of Ohio Environmental Protection Agency

Manual of Ohio EPA

Sludge Program Field Sampling Methods

And

Quality Assurance Practices

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Division of Surface Water

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Part I: Soil and Soil/Sludge Sampling

The type of sampling and analyses are dependent upon factors such as the volume of sludge produced annually, the type of treatment performed on the sludge, the final use or disposal of the sludge, and the sludge parameter(s) of concern.

A. Equipment Preparation

A soil sample is best taken with a soil probe or an auger. These tools help to ensure that an equal amount of soil sample/subsample will be collected to a definite depth at the sampling site. A spade, a knife, or a trowel can be used to collect thin slices or sections of soil samples/subsamples. A spade, a knife, or a trowel can also be used to remove soil from a probe or auger, to break up cores, or to mix grab subsamples for a composite sample so that any composite subsample removed after mixing is a homogeneous mixture.

Proper equipment decontamination protocol must be used to ensure that samples are free from cross contamination. Equipment must be washed with a phosphorus free detergent, rinsed with distilled water and dried. If a sample must be collected and the on-site washing of equipment is not possible, push the probe, or use the auger, several times at the new site and remove any material adhering to the equipment prior to collecting the samples at the new site.

Soil samples should be collected and placed in a clean plastic pail or box. Individual grab samples must be thoroughly mixed to provide a homogenous composite subsample when composite samples are collected.

B. Sample Collection Procedure

Soil samples can be collected at any time. However, samples collected to analyze for nutrients or metals should be collected prior to spring planting. Soil samples collected to analyze for other chemicals (e.g., organic chemicals of concern) should be collected after sludge has been applied to a site. A control (background) uncontaminated sample must be collected for chemical analyses when chemicals of concern are being evaluated. Collectors must wear disposable gloves when collecting soil and soil/sludge samples.

A soil probe, an auger, a spade, or a garden trowel can be used to collect a soil sample. A plastic bucket should be used to collect soil subsamples/samples.

Each composite sampling should represent an area of from 15 to 20 uniform acres. The site should be uniform in soil series, slope, drainage and erosion, and fertilizer application, including sewage sludge, with less than 5 acres of rolling land. In general, any area that is large enough to spread sludge separately should be sampled separately.

Establish a transect across the widest portion of the field and collect grab subsamples every 75 to 100 feet; collect a minimum of 15 grab subsamples per 5 acres. Low spots or other unusual or atypical areas (e.g., a sludge stockpiling location, or an area where lime or fertilizer was spilled) must be omitted from the sample, or sampled separately. Repeat the collection procedure on two adjacent parallel transects, each located one-quarter of the distance between the first, longest transect and the field boundary. Repeat the entire sampling procedure by sampling three transects perpendicular to the first three transects sampled. Using this procedure, a total of six transects will be sampled.

Samples should be taken to plow depth, or approximately to an 8 inch depth. Samples should be collected between rows where row crop acreage is being sampled. Samples should be collected to the rooting zone of from 3 to 4 inches where pasture grass acreage is being sampled. All grab subsamples must be broken up and mixed thoroughly to homogenize the mixture before a composite subsample is removed. The sample should be transferred to an appropriate container (Appendix1). Sample weight must be at least 10 grams for metals, 10 grams for mercury, 10 grams for nutrients (approximately 100 grams for all inorganic parameter analyses), and 100 grams for organic analyses. Sediment samples are not preserved. Samples are kept at 4 degrees C.

Analyte sample concentration for sludge and sludge/soil samples is reported as parameter concentration per unit dry weight. Composite samples provide an average parameter concentration.

C. Sample Documentation

Sample labels must include the following information:

1. Sampling organization's name (e.g., DSW),
2. Facility or location name being sampled,
3. Collector(s)
4. Type of sample (i.e., grab, 24 hour composite, etc.),
5. Date and military time (record time as 24 hour time, e.g., 1:00 P.M. is 1300),

6. Specific sample location (Latitude and Longitude recorded in decimal degrees – The method used to determine Latitude and Longitude must be identified), and
7. Sample container number.

A field note book should include all of the above sample information, parameters analyzed and any special remarks. A sketch showing the location sampled and an indication where the sample(s) was (were) collected should be included.

D. Chain-of-Custody

All samples submitted for analyses must include a Chain-of-Custody record. A Chain-of-Custody provides a record of sample transfer from person to person. This document helps to protect the integrity of the sample by ensuring that only authorized persons have custody of the sample. The Chain-of-Custody procedure ensures that an enforceable record of sample transfer has occurred, which is necessary if the sample results are to be used in a judicial proceeding alleging violations of sludge standards. A Chain-of-Custody record for each sample's collection and handling history from the time of sample collection, sample transport and sample receipt by the laboratory includes the information listed on the sample container. All personnel handling the sample must sign, date and note the time of day (military time) on the Chain-of-Custody sheet.

E. Laboratory Coordination

All sampling must be coordinated with the laboratory. The laboratory (DES for the DSW) must be contacted to ensure proper preparation of the sample(s), required forms and labeling procedures, and transportation and the drop off procedure.

Analytical parameters, analytical methods, maximum holding times, sample container type and comments are listed in Appendix 1. Sludge, sediment and soil samples are not preserved, and are kept at 4 degrees C. Samples are prepared using Ohio EPA Method 400.9 (microwave preparation) using 0.5 to 0.65 g (wet weight) of sample for all analytical procedures for total metals identified, U.S. EPA Method 200.7 and U.S. EPA Method 200.8 (ICP and ICP/MS).

Method SW-846 Method 3050 (using an equivalent to 1g dry weight) for any of the analytical procedures identified when analyzing for Total Metals (Total Arsenic, Total Cadmium, Total Copper, Total Lead, Total Molybdenum, Total Nickel, Total Potassium, Total Selenium and Total Zinc) will be used if requested.

Analytical Method SW-846 is used for DERR samples only. Analytical Method SW-846 will be used for DSW analyses if requested.

Use SW-846 Method 7471 to analyze for Total Mercury in semisolid and solid wastes. The digestion method is contained in the analytical method procedure.

The procedure for determining Specific Oxygen Uptake Rate (SOUR) in samples is very sensitive to sample temperature variation and lag time between sample collection and test initiation. Replicate samples should be taken. SOUR must be calculated based upon the total solids dry weight rather than on volatile solids.

Aspiration analyses of metals are applicable at moderate concentrations in clean, complex matrix systems. Inductively Coupled Plasma (ICP) methods are applicable over a broad concentration range and are especially sensitive for refectory elements. Detection limits for AA Furnace methods are generally lower than for ICP methods. The analytical method selected will be determined by sample type and sampling objective(s). Select an analytical procedure with a laboratory Reporting Limit (RL) below the analyte concentration objective. Analyte concentrations are reported as concentration per unit dry weight. Refer to DES' WEB site for analytical Method Detection Limits (MDLs) and Laboratory Reporting Limits (RLs).

F. References (additional information)

DSW Permit Guidance 11, detail testing requirements for sewage sludge for land application.

Congressional Federal Registrar (CFR) 40 CFR Part 503.

Ohio Administrative Code (OAC), Chapter 3745-40.

U.S. EPA. 1989. "POTW Sludge Sampling and Analysis Guidance Document," August 1989; for U.S. EPA; by Science Applications International Corporation.

U.S. EPA. 1996. "Test Methods for Evaluation Solid Waste (SW-846), Update III", December 1996. U.S. EPA, Office of Solid Waste and Emergency Response.

U.S. EPA. 2003. "Control of Pathogens and Vector Attraction in Sewage Sludge, Revised July 2003. This document is also known as the "White House Document".

Appendix 1

Table 1: Parameter, analytical method, maximum holding time and comments for soil and soil/sludge sampling (a)

A. Physical Characteristics, Total Nutrients and Dioxin

Parameter	Analytical Method	Holding Time Container	Comments
pH	SW-9045	Immediate Plastic or glass	The sample is mixed with a prescribed liquid and the pH is determined with a probe. Record pH at 25C, or correct for temperature and report at 25C
Conductivity	SW-9050	28 days Cool to 4C Plastic or glass	Record conductivity at 25C, or correct for temperature and report results for 25C
Total Solids	SM-2540 G	7 Days Cool to 4C Plastic or glass	SM-2540 is the recommended Method for solid and semi-solid samples.
Volatile Solids	SM-2540 G	7 Days Cool to 4C Plastic or glass	SM-2540 is the recommended Method for solid and semi-solid samples.
Specific Oxygen Uptake Rate (SOUR)	SM-2710 See Appendix D EPA/625/R-92/013 October 1999	Perform as soon as possible Plastic or glass	See text
Soil Phosphorus (P)	Bray/Kurtz Test for P Mehlich 3 Test for P Olsen (NaHCO3)P	28 days Cool to 4C 500ml amber jar	North Central Regional Research, Pub. No. 221 (Revised): Recommended Chemical Soil test
Total Kjeldahl Nitrogen (TKN)	SM-4500-N organic	28 days Cool to 4C Plastic or glass	TKN nitrogen is the sum of organic and ammonia nitrogen. Sample digestion and distillation are required. Include or reference in method

a. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: A. Physical Characteristics, Total Nutrients and Dioxin (continued)

Parameter	Analytical Method	Holding Time Container	Comments
Total Ammonia Nitrogen (NH3-N)	SM-4500-NH3	7 days Cool to 4C Plastic or glass	All samples must be distilled using procedure SM-4500-NH3 B prior to using a listed specific analytical method.
Total Nitrate Nitrogen (NO3-N)	SM-4500-NO3 SW-846 Method 9056 SW-846 Method 9210	28 days Cool to 4C Plastic or glass	Nitrate nitrogen is the fully oxidized state of nitrogen. The presence of organics may interfere with the method.
Total Phosphorus (T-P)	SM-4500-P	7 days Cool to 4C Plastic or glass	Pay close attention to the sample preparation requirements found in section 4500-P-B.
Dioxin TTEs	U.S. EPA Method 1613B U.S. EPA Method 1668A	30 days Cool to 4C Amber glass Teflon lined lid	For liquid or cake samples A cake sample is preferred
Organic Chemical of Concern	GC-MS Method SW-846	14 days Cool to 4C Amber glass Teflon lined lid	

a. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: B Total Metals (a,b)

Parameter	Analytical Method	Holding Time Container	Comments
Arsenic (T-As)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Cadmium (T-Cd)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Copper (T-Cu)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Lead (T-Pb)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Molybdenum (T-Mo)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Nickel (T-Ni)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Potassium (T-K)	USEPA 200.7 ICP	6 months Cool to 4C Plastic or glass	See text

a. Ohio EPA sample microwave preparation SOP 400.9 is used for sludge, sediment and soil metals parameters.

b. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: B Total Metals (continued)

Parameter	Analytical Method	Holding Time Container	Comments
Selenium (T-Se)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Zinc (T-Zn)	USEPA 200.7 ICP	6 months Cool to 4C Plastic or glass	See text
Mercury (T-Hg)	Cold Vapor (manual) SW-846 Method 7470 SW-846 Method 7471 AA Furnace SW-846 ICP SW-846 Method 6010 US EPA Method 245.1	28 days Cool to 4C Plastic or glass	Use SW-846 Method 7470 for liquid wastes. Use SW-846 Method 7471 for solid and semisolid wastes. The digestion procedure is included in the analytical method.

a. Ohio EPA sample microwave preparation SOP 400.9 is used for sludge, sediment and soil metals parameters.

b. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: C. Organisms

Parameter Organism	Analytical Method	Holding Time Container	Comments
Fecal Coliform	SM-9221 E Most Probable Number Required for Class A and recommended for Class B, Alternative 1 SM-9222 D Membrane filter	24 Hours Cool to 4C Plastic or glass	Both procedures are very temperature sensitive. The samples must be analyzed within the defined holding times.
Salmonella sp.	Kenner and Clark See Appendix G EPA/625/R-92/013 October 1999 SM-9260 D	24 Hours Cool to 4C Plastic or glass	Large sample volumes are needed due to low concentrations of salmonella in wastewater and sludge. More than one procedure may be necessary to determine salmonella presence because of the large number of salmonella species.
Enteric Viruses	ASTM Method D 4994-89	48 Hours Up to 2 weeks Cool at 0F Plastic or glass	Concentration of the sample is necessary due to the presumably low numbers of viruses in the sample.
Helminth Ova	Yanko See Appendix I EPA/625/R-92/013 October 1999	1 Month Cool to 4C Plastic or glass	The analyst must be familiar with other ova test methods which are found in the same document. All ova identified are considered viable ova.

Part II: Liquid Sludge Sampling

A. Equipment Preparation

A liquid sludge sample is best taken as sludge is being transferred from one vessel to another. This should be done downstream from a pump that serves to mix the sewage sludge thoroughly. Other in-vessel sampling options include using a core sampler, a Sludge Judge, or a sludge nabber.

Proper equipment decontamination protocol must be used to ensure that samples are free from cross contamination. Equipment must be washed with a phosphorus free detergent, rinsed with distilled water and dried. Collect three timed, composite subsamples of equal volume from the top, middle and bottom of the vessel being sampled. Collect a minimum of 12 subsamples per vessel; four subsamples from each vessel level for each of the three timed collections.

Collectors must wear disposable gloves when collecting liquid sludge samples. Liquid sludge samples should be collected and placed in a clean plastic pail and thoroughly mixed to provide a homogenous composite subsample.

B. Sample Collection Procedure for Liquid Sludge Being Transferred

1. Anaerobically Digested Sludge

The sample should be collected on the discharge side of the positive displacement pumps.

2. Aerobically Digested Sludge

The sample should be collected on the discharge side of the positive displacement pumps directly from the digester if batch digestion is used.

Two cautions are in order for collecting aerobically digested sludge. Volatile organic compounds may purge with escaping air when air entrains in the sample. And, solids separate rapidly in well-digested sludge when aeration is shut off.

3. Thickened Sludge

The sample should be collected on the discharge side of the positive displacement pumps.

4. Heat Treatment

The sample should be collected on the discharge side of the positive displacement pumps after decanting. Care should be taken when sampling heat treated sludge. There is a high tendency for solids separation, and the high temperature of the sample (>600C as sampled) can cause problems with some sample containers due to cooling and subsequent concentration of entrained gases.

For sludge gathered by a Sludge Judge, lower the Sludge Judge to the bottom of the tank. When the bottom has been reached and the pipe has been filled to the surface level, tug slightly on the rope as the unit is raised. This sets the check valve which traps the column of solids and liquid in the Sludge Judge.

For sludge samples collected with a sludge nabber, section the sample space into decision unit areas (e.g., quadrants, clock face, etc.). Composite equal amounts of bottom, middle and top sludge from a minimum of 3 areas of each decision unit area for a total of 12 grab samples.

Sediment samples are not preserved. Samples are kept at 4 degrees C. A composite sample should contain equal amounts of bottom, middle and top levels of sludge. A composite sample should produce an average parameter concentration. Percent solids are used to determine analyte concentrations reported as dry weight concentrations.

C. Sample Documentation

Sample labels must include the following information:

1. Sampling organization's name (e.g., DSW),
2. Facility or location name being sampled,
3. Collector(s)
4. Type of sample (i.e., 24 hour grab, composite, etc.),
5. Date and military time (record time as 24 hour time, e.g., 1:00 P.M. is 1300),
6. Specific sample location (Latitude and Longitude recorded in decimal degrees – The method used to determine Latitude and Longitude must be identified), and
7. Sample container number.

A field note book should include all of the above sample information, parameters analyzed and any special remarks. A sketch showing the location sampled and an indication where the sample(s) was (were) collected should be included.

D. Chain-of-Custody

All samples submitted for analyses must include a Chain-of-Custody record. A Chain-of-Custody provides a record of sample transfer from person to person. This document helps to protect the integrity of the sample by ensuring that only authorized persons have custody of the sample. The Chain-of-Custody procedure ensures that an enforceable record of sample transfer has occurred, which is necessary if the sample results are to be used in a judicial proceeding alleging violations of sludge standards. A Chain-of-Custody record for each sample's collection and handling history from the time of sample collection, sample transport and sample receipt by the laboratory includes the information listed on the sample container. All personnel handling the sample must sign, date and note the time of day (military time) on the Chain-of-Custody sheet.

E. Laboratory Coordination

All sampling must be coordinated with the laboratory. The laboratory (DES for the DSW) must be contacted to ensure proper preparation of the sample(s), required forms and labeling procedures and transportation and the drop off procedure.

Analytical parameters, analytical methods, maximum holding times, sample container type and comments are listed in Appendix 1. Sludge, sediment and soil samples are not preserved. Samples are prepared using Ohio EPA Method 400.9 (microwave preparation) using 0.5 to 0.65 g (wet weight) of sample for all analytical procedures for total metals identified, U.S. EPA Method 200.7, and U.S. EPA Method 200.8 (ICP and ICP/MS).

Method SW-846 Method 3050 (using an equivalent to 1g dry weight) for any of the analytical procedures identified when analyzing for Total Metals (Total Arsenic, Total Cadmium, Total Copper, Total Lead, Total Molybdenum, Total Nickel, Total Potassium, Total Selenium and Total Zinc) will be used if requested.

Analytical Method SW-846 is used for DERR samples only. Analytical Method SW-846 will be used for DSW analyses if requested.

The analytical method selected will be determined by sample type and sampling objective(s). Select an analytical procedure with a laboratory Reporting Limit (RL) below the analyte concentration objective. Analyte concentrations are reported as concentration per unit dry weight. Refer to DES' WEB site for analytical Method Detection Limits (MDLs) and Laboratory Reporting Limits (RLs).

Use SW-846 Method 7470 to analyze for Total Mercury in liquid wastes. The digestion method is contained in the analytical method procedure.

The procedure for determining Specific Oxygen Uptake Rate (SOUR) in samples is very sensitive to sample temperature variation and lag time between sample collection and test initiation. Replicate samples should be taken. SOUR must be calculated based upon the total solids dry weight rather than on volatile solids.

F. References (additional information)

DSW Permit Guidance 11, detail testing requirements for sewage sludge for land application.

Congressional Federal Registrar (CFR) 40 CFR Part 503.

Ohio Administrative Code (OAC), Chapter 3745-40.

U.S. EPA. 1989. "POTW Sludge Sampling and Analysis Guidance Document," August 1989; for U.S. EPA; by Science Applications International Corporation.

U.S. EPA. 1996. "Test Methods for Evaluation Solid Waste (SW-846), Update III", December 1996. U.S. EPA, Office of Solid Waste and Emergency Response.

U.S. EPA. 2003. "Control of Pathogens and Vector Attraction in Sewage Sludge, Revised July 2003. This document is also known as the "White House Document".

Appendix 1

Table 1: Parameter, analytical method, maximum holding time and comments for soil and soil/sludge sampling (a)

A. Physical Characteristics, Total Nutrients and Dioxin

Parameter	Analytical Method	Holding Time Container	Comments
pH	SW-9045	Immediate Plastic or glass	The sample is mixed with a prescribed liquid and the pH is determined with a probe. Record pH at 25C, or correct for temperature and report at 25C
Conductivity	SW-9050	28 days Cool to 4C Plastic or glass	Record conductivity at 25C, or correct for temperature and report results for 25C
Total Solids	SM-2540 G	7 Days Cool to 4C Plastic or glass	SM-2540 is the recommended Method for solid and semi-solid samples.
Volatile Solids	SM-2540 G	7 Days Cool to 4C Plastic or glass	SM-2540 is the recommended Method for solid and semi-solid samples.
Specific Oxygen Uptake Rate (SOUR)	SM-2710 See Appendix D EPA/625/R-92/013 October 1999	Perform as soon as possible Plastic or glass	See text
Soil Phosphorus (P)	Bray/Kurtz Test for P Mehlich 3 Test for P Olsen (NaHCO3)P	28 days Cool to 4C 500ml amber jar	North Central Regional Research, Pub. No. 221 (Revised): Recommended Chemical Soil test
Total Kjeldahl Nitrogen (TKN)	SM-4500-N organic	28 days Cool to 4C Plastic or glass	TKN nitrogen is the sum of organic and ammonia nitrogen. Sample digestion and distillation are required. Include or reference in method

a. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: A. Physical Characteristics, Total Nutrients and Dioxin (continued)

Parameter	Analytical Method	Holding Time Container	Comments
Total Ammonia Nitrogen (NH3-N)	SM-4500-NH3	7 days Cool to 4C Plastic or glass	All samples must be distilled using procedure SM-4500-NH3 B prior to using a listed specific analytical method.
Total Nitrate Nitrogen (NO3-N)	SM-4500-NO3 SW-846 Method 9056 SW-846 Method 9210	28 days Cool to 4C Plastic or glass	Nitrate nitrogen is the fully oxidized state of nitrogen. The presence of organics may interfere with the method.
Total Phosphorus (T-P)	SM-4500-P	7 days Cool to 4C Plastic or glass	Pay close attention to the sample preparation requirements found in section 4500-P-B.
Dioxin TTEs	U.S. EPA Method 1613B U.S. EPA Method 1668A	30 days Cool to 4C Amber glass Teflon lined lid	For liquid or cake samples A cake sample is preferred
Organic Chemical of Concern	GC-MS Method SW-846	14 days Cool to 4C Amber glass Teflon lined lid	

a. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: B Total Metals (a,b)

Parameter	Analytical Method	Holding Time Container	Comments
Arsenic (T-As)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Cadmium (T-Cd)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Copper (T-Cu)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Lead (T-Pb)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Molybdenum (T-Mo)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Nickel (T-Ni)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Potassium (T-K)	USEPA 200.7 ICP	6 months Cool to 4C Plastic or glass	See text

a. Ohio EPA sample microwave preparation SOP 400.9 is used for liquid sludge metals parameters.

b. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: B Total Metals (continued)

Parameter	Analytical Method	Holding Time Container	Comments
Selenium (T-Se)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Zinc (T-Zn)	USEPA 200.7 ICP	6 months Cool to 4C Plastic or glass	See text
Mercury (T-Hg)	Cold Vapor (manual) SW-846 Method 7470 SW-846 Method 7471 AA Furnace SW-846 ICP SW-846 Method 6010 U.S. EPA Method 245.1	28 days Cool to 4C Plastic or glass	Use SW-846 Method 7470 for liquid wastes. Use SW-846 Method 7471 for solid and semisolid wastes. The digestion procedure is included in the analytical method.

a. Ohio EPA sample microwave preparation SOP 400.9 is used for liquid sludge metals parameters.

b. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: C. Organisms

Parameter Organism	Analytical Method	Holding Time Container	Comments
Fecal Coliform	SM-9221 E Most Probable Number Required for Class A and recommended for Class B, Alternative 1 SM-9222 D Membrane filter	24 Hours Cool to 4C Plastic or glass	Both procedures are very temperature sensitive. The samples must be analyzed within the defined holding times.
Salmonella sp.	Kenner and Clark See Appendix G EPA/625/R-92/013 October 1999 SM-9260 D	24 Hours Cool to 4C Plastic or glass	Large sample volumes are needed due to low concentrations of salmonella in wastewater and sludge. More than one procedure may be necessary to determine salmonella presence because of the large number of salmonella species.
Enteric Viruses	ASTM Method D 4994-89	48 Hours Up to 2 weeks Cool at 0F Plastic or glass	Concentration of the sample is necessary due to the presumably low numbers of viruses in the sample.
Helminth Ova	Yanko See Appendix I EPA/625/R-92/013 October 1999	1 Month Cool to 4C Plastic or glass	The analyst must be familiar with other ova test methods which are found in the same document. All ova identified are considered viable ova.

Part III: Cake Sludge Sampling

A. Equipment Preparation

A cake sludge sample is best taken as sludge is being transferred, usually on conveyors. If this is not possible, a stockpile sample can be taken using an auger, a probe, or a shovel. These tools will help to ensure that all depths of the stockpile are represented.

Collectors must wear disposable gloves when collecting cake sludge samples. Cake sludge subsamples should be collected and placed in a clean plastic pail or box. Individual grab samples must be thoroughly mixed to provide a homogenous composite subsample when composite samples are collected.

Proper equipment decontamination protocol must be used to ensure that samples are free from cross contamination. Equipment must be washed with a phosphorus free detergent, rinsed with distilled water and dried. Sampling should composite three subsamples of equal volume from top, middle and bottom of the vessel being sampled for a total of 12 grab samples.

B. Sample Collection Procedure for Cake Sludge Being Transferred

1. Dried, Composted, or Thermally Reduced

Collect a composite sample by **s**ampling the sludge from the material collection conveyors and bulk containers. Collected an equal volume of sample material from the top, middle and bottom of all vessels being sampled; collect four subsamples of the sludge mass at the three depths at four locations. Composite 12 grab subsamples taken at various locations from the conveyor.

2. Dewatered: Belt Filter Press, Centrifuge, or Vacuum Filter Press

Collect a composite sample of the sludge from the discharge chute.

3. Press: Plate or Frame

Collect a composite sample of the sludge from the storage bin. Select four locations within the storage bin and collect an equal amount of material from each location. Combine the four subsamples into a composite sample.

4. Drying Beds

Divide the bed into quarters and grab equal amounts of material from the center of each quarter. Each subsample should include the entire depth of the sludge down to the sand. An equal volume of sampled material from top, middle and bottom of the vessel being sampled should be collected. Combine the four subsamples into a composite sample.

5. Composite Piles

Collect a composite sample of the sludge directly from the front end loader as the sludge is being loaded into trucks to be hauled away or stored.

Sediment samples are not preserved. Samples are kept at 4 degrees C. Sediment samples are not preserved. Samples are kept at 4 degrees C. A composite sample should produce an average parameter concentration. Analyte sample concentration for sludge cake is reported as parameter concentration per unit dry weight.

C. Sample Documentation

Sample labels must include the following information:

1. Sampling organization's name (e.g., DSW),
2. Facility or location name being sampled,
3. Collector(s)
4. Type of sample (i.e., 24 hour grab, composite, etc.),
5. Date and military time (record time as 24 hour time, e.g., 1:00 P.M. is 1300),
6. Specific sample location (Latitude and Longitude recorded in decimal degrees – The method used to determine Latitude and Longitude must be identified), and
7. Sample container number.

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A field note book should include all of the above sample information, parameters analyzed and any special remarks. A sketch showing the location sampled and an indication where the sample(s) was (were) collected should be included.

D. Chain-of-Custody

All samples submitted for analyses must include a Chain-of-Custody record. A Chain-of-Custody provides a record of sample transfer from person to person. This document helps to protect the integrity of the sample by ensuring that only authorized persons have custody of the sample. The Chain-of-Custody procedure ensures that an enforceable record of sample transfer has occurred, which is necessary if the sample results are to be used in a judicial proceeding alleging violations of sludge standards. A Chain-of-Custody record for each sample's collection and handling history from the time of sample collection, sample transport and sample receipt by the laboratory includes the information listed on the sample container. All personnel handling the sample must sign, date and note the time of day (military time) on the Chain-of-Custody sheet.

E. Laboratory Coordination

All sampling must be coordinated with the laboratory. The laboratory (DES for the DSW) must be contacted to ensure proper preparation of the sample(s), required forms and labeling procedures and transportation and the drop off procedure.

Analytical parameters, analytical methods, maximum holding times, sample container type and comments are listed in Appendix 1. Sludge, sediment and soil samples are not preserved. Samples are prepared using Samples are prepared using Ohio EPA Method 400.9 Samples are prepared using Ohio EPA Method 400.9 (microwave preparation) using 0.5 to 0.65 g (wet weight) of sample for all analytical procedures for total metals identified, U.S. EPA Method 200.7 and U.S. EPA Mehtod 200.8 (ICP and ICP/MS).

Method SW-846 Method 3050 (using an equivalent to 1g dry weight) for any of the analytical procedures identified when analyzing for Total Metals (Total Arsenic, Total Cadmium, Total Copper, Total Lead, Total Molybdenum, Total Nickel, Total Potassium, Total Selenium and Total Zinc) will be used if requested.

Inductively Coupled Plasma (ICP) methods are applicable over a broad concentration range and are especially sensitive for refectory elements. The analytical method selected will be determined by sample type and sampling objective(s). If possible, select an analytical procedure with a laboratory Reporting Limit (RL) below the analyte concentration objective. Analyte concentrations are reported as concentration per unit dry weight. Refer to DES' WEB site for analytical Method Detection Limits (MDLs) and Laboratory RLs.

Analytical Method SW-846 is used for DERR samples only. Analytical Method SW-846 will be used for DSW analyses if requested.

Use SW-846 Method 7471 to analyze for Total Mercury in semisolid and solid wastes. The digestion method is contained in the analytical method procedure.

The procedure for determining Specific Oxygen Uptake Rate (SOUR) in samples is very sensitive to sample temperature variation and lag time between sample collection and test initiation. Replicate samples should be taken. SOUR must be calculated based upon the total solids dry weight rather than on volatile solids.

F. References (additional information)

DSW Permit Guidance 11, detail testing requirements for sewage sludge for land application.

Congressional Federal Registrar (CFR) 40 CFR Part 503.

Ohio Administrative Code (OAC), Chapter 3745-40.

U.S. EPA. 1989. "POTW Sludge Sampling and Analysis Guidance Document," August 1989; for U.S. EPA; by Science Applications International Corporation.

U.S. EPA. 1996. "Test Methods for Evaluation Solid Waste (SW-846), Update III", December 1996. U.S. EPA, Office of Solid Waste and Emergency Response.

U.S. EPA. 1999. Environmental Regulations and Technology Control of Pathogens and Vector Attraction in Sewage Sludge. Appendix G, Kenner and Clark. EPA/625/R-92/013, October 1999.

U.S. EPA. 2003. "Control of Pathogens and Vector Attraction in Sewage Sludge, Revised July 2003. This document is also known as the "White House Document".

Appendix 1

Table 1: Parameter, analytical method, maximum holding time and comments for cake sludge sampling (a)

A. Physical Characteristics, Total Nutrients and Dioxin

Parameter	Analytical Method	Holding Time Container	Comments
pH	SW-9045	Immediate Plastic or glass	The sample is mixed with a prescribed liquid and the pH is determined with a probe. Record pH at 25C, or correct for temperature and report at 25C
Conductivity	SW-9050	28 days Cool to 4C Plastic or glass	Record conductivity at 25C, or correct for temperature and report results for 25C
Total Solids	SM-2540 G	7 Days Cool to 4C Plastic or glass	SM-2540 is the recommended Method for solid and semi-solid samples.
Volatile Solids	SM-2540 G	7 Days Cool to 4C Plastic or glass	SM-2540 is the recommended Method for solid and semi-solid samples.
Specific Oxygen Uptake Rate (SOUR)	SM-2710 See Appendix D EPA/625/R-92/013 October 1999	Perform as soon as possible Plastic or glass	See text
Soil Phosphorus (P)	Bray/Kurtz Test for P Mehlich 3 Test for P Olsen (NaHCO3)P	28 days Cool to 4C 500ml amber jar	North Central Regional Research, Pub. No. 221 (Revised): Recommended Chemical Soil test
Total Kjeldahl Nitrogen (TKN)	SM-4500-N organic	28 days Cool to 4C Plastic or glass	TKN nitrogen is the sum of organic and ammonia nitrogen. Sample digestion and distillation are required. Include or reference in method

a. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: A. Physical Characteristics, Total Nutrients and Dioxin (continued)

Parameter	Analytical Method	Holding Time Container	Comments
Total Ammonia Nitrogen (NH3-N)	SM-4500-NH3	7 days Cool to 4C Plastic or glass	All samples must be distilled using procedure SM-4500-NH3 B prior to using a listed specific analytical method.
Total Nitrate Nitrogen (NO3-N)	SM-4500-NO3 SW-846 Method 9056 SW-846 Method 9210	28 days Cool to 4C Plastic or glass	Nitrate nitrogen is the fully oxidized state of nitrogen. The presence of organics may interfere with the method.
Total Phosphorus (T-P)	SM-4500-P	7 days Cool to 4C Plastic or glass	Pay close attention to the sample preparation requirements found in section 4500-P-B.
Dioxin TTEs	U.S. EPA Method 1613B U.S. EPA Method 1668A	30 days Cool to 4C Amber glass Teflon lined lid	For liquid or cake samples A cake sample is preferred
Organic Chemical of Concern	GC-MS Method SW-846	14 days Cool to 4C Amber glass Teflon lined lid	

a. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: B Total Metals

Parameter	Analytical Method	Holding Time Container	Comments
Arsenic (T-As)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Cadmium (T-Cd)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Copper (T-Cu)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Lead (T-Pb)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Molybdenum (T-Mo)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Nickel (T-Ni)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Potassium (T-K)	USEPA 200.7 ICP	6 months Cool to 4C Plastic or glass	See text

a. Ohio EPA sample microwave preparation SOP 400.9 is used for cake sludge metals parameters.

b. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: B Total Metals (continued)

Parameter	Analytical Method	Holding Time Container	Comments
Selenium (T-Se)	USEPA 200.7 ICP USEPA 200.8 ICP/MS	6 months Cool to 4C Plastic or glass	See text
Zinc (T-Zn)	USEPA 200.7 ICP	6 months Cool to 4C Plastic or glass	See text
Mercury (T-Hg)	Cold Vapor (manual) SW-846 Method 7470 SW-846 Method 7471 AA Furnace SW-846 ICP SW-846 Method 6010 U.S. EPA Method 245.1	28 days Cool to 4C Plastic or glass	Use SW-846 Method 7470 for liquid wastes. Use SW-846 Method 7471 for solid and semisolid wastes. The digestion procedure is included in the analytical method.

a. Ohio EPA sample microwave preparation SOP 400.9 is used for cake sludge metals parameters.

b. Method SW846 (Methods 6010B for ICP parameters and Method 6020A for ICP/MS parameters) are used if requested.

Table 1: C. Organisms

Parameter Organism	Analytical Method	Holding Time Container	Comments
Fecal Coliform	SM-9221 E Most Probable Number Required for Class A and recommended for Class B, Alternative 1 SM-9222 D Membrane filter	24 Hours Cool to 4C Plastic or glass	Both procedures are very temperature sensitive. The samples must be analyzed within the defined holding times.
Salmonella sp.	Kenner and Clark See Appendix G EPA/625/R-92/013 October 1999 SM-9260 D	24 Hours Cool to 4C Plastic or glass	Large sample volumes are needed due to low concentrations of salmonella in wastewater and sludge. More than one procedure may be necessary to determine salmonella presence because of the large number of salmonella species.
Enteric Viruses	ASTM Method D 4994-89	48 Hours Up to 2 weeks Cool at 0F Plastic or glass	Concentration of the sample is necessary due to the presumably low numbers of viruses in the sample.
Helminth Ova	Yanko See Appendix I EPA/625/R-92/013 October 1999	1 Month Cool to 4C Plastic or glass	The analyst must be familiar with other ova test methods which are found in the same document. All ova identified are considered viable ova.

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