

## APPENDIX H

### EXAMPLE OF SLUDGE SAMPLING PROCEDURE

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

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

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

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## SLUDGE SAMPLING STANDARD OPERATING PROCEDURE

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1. A week to several days prior to the proposed sampling, confirm sludge processing (dewatering and treatment) to ensure that sludge in the appropriate form (liquid versus dewatered, untreated cake versus treated biosolids) will be available for sampling at the proposed date, time, and sampling point.
2. A week to several days prior to the proposed sampling date, confirm that the contract laboratory performing the analyses is prepared to accept samples on the proposed sampling date.
3. At least one day before collecting samples, assemble the sampling equipment. Ensure that all equipment is clean and in good working order (See attached checklist and cleaning procedure).
4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.
5. After arrival at the sampling location/sampling point (as determined in the sampling plan), evaluate the operation of the sludge handling train (dewatering, biosolids treatment, etc.). Any observable deviations from normal operation should be noted prior to collecting samples.
6. Put on nitrile gloves and any other required/desired personal safety equipment.
7. Collect the first grab sample of the 8 grab samples that will make up the composite and record the time. Using a 500 mL glass beaker and a stainless steel trowel, collect the sample from the belt filter press as the sludge falls into the roll-off container. The first grab sample and all remaining grabs should be approximately equal in volume (~ 200 mL). Do not forget to collect any required field duplicates or blanks.
8. After the first grab has been collected, it should be placed in a stainless steel bucket. A plastic syringe with the luer lock end removed is used to collect a zero-headspace subsample of about 5 mL in volume from the original grab sample. This subsample should be placed in a 40 mL glass vial filled with 10 mL of methanol preservative. This sample should be placed on ice and cooled to 4° C until analyzed according to EPA Method 8260 for volatile organic compounds (VOC).
9. After the first grab sample, another grab sample should be collected every 30 minutes and placed in the stainless steel bucket until all 8 grab samples have been collected. Again, the grab samples should be of approximately equal size (weight or volume). During the time between samples, the stainless steel bucket should be covered and placed on ice or refrigerated. (This is necessary whenever the interval between grab samples is longer than five minutes.) The time of collection of the last grab sample should be recorded.

## Appendix H: Example of Sludge Sampling Procedure

10. Upon collection of the last grab sample, thoroughly mix all material accumulated in the stainless steel bucket with a stainless steel trowel. The goal of the mixing process is to produce a homogeneous sample.
11. After mixing, label all sample containers with the following information:
  - a) Sample identification number (ID)
  - b) Date and time of collection
  - c) Sample location
  - d) Person collecting sample
  - e) Preservative
  - f) Required test(s)
12. After labeling, fill each sample container with portions of the homogenized material in the stainless steel bucket.
13. After each sample container is filled, seal it with a signed custody seal and place it on ice in a cooler for transportation to the laboratory.
14. Prior to delivering the samples to the lab, complete a chain-of-custody sheet to document proper sample handling.
15. After sample delivery, clean all equipment according to established procedures and store in a clean, dry area.

## EQUIPMENT CHECKLIST

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- 1) Protective Gear
  - a. Nitrile gloves
  - b. Tyvek sleeves
- 2) Sample handling and collection
  - a. Stainless steel bucket
  - b. 500 mL glass or Teflon beaker
  - c. Stainless steel trowel
- 3) Transporting and preservation
  - a. Sample containers
    - 1) VOC - 40 mL glass vial with 10 mL of methanol preservative
    - 2) SVOC – 250 mL, wide-mouth, amber glass jar
    - 3) Pesticides and PCBs – 250 mL, wide-mouth, amber glass jar
    - 4) Dioxin – 125 mL, wide-mouth, amber glass jar
    - 5) Metals and nutrients – 500 mL, wide-mouth, clear glass jar
  - b. Sample cooler with ice
- 4) Sample ID and Documentation
  - a. Markers and pens
  - b. Sample container labels
  - c. Custody seals
  - d. Chain of custody/sample submittal form
  - e. Field notebook/ sample log/field data sheet
- 5) Cleaning equipment
  - a. Disposable towels
  - b. Soap
  - c. Scrub brush
  - d. Rinse water
  - e. Deionized water
  - d. 10% hydrochloric acid solution
  - e. Rinse water
  - f. Deionized water
  - g. Aluminum foil or plastic wrap

## EQUIPMENT PREPARATION AND CLEANING PROCEDURE

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

- 1) Rinse equipment with warm tap water to remove the majority of solids.
- 2) Using a brush and a low-phosphate lab detergent, scrub the equipment to remove all residues.
- 3) After scrubbing, rinse the equipment three times with tap water.
- 4) Next, rinse the equipment with a 10% hydrochloric acid solution – allow at least 30 seconds of contact time.
- 5) Perform a final rinse, which should be a triple rinse with deionized water.
- 6) After cleaning, allow the equipment to air-dry. To store, cover beakers and buckets with clean aluminum foil or plastic wrap. Sampling implements should also be wrapped in foil or plastic wrap to keep them clean while in storage.

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