

NPDES Compliance Inspection Manual

Appendix AM



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Appendix AM – Sampling Procedures and Equipment

SAMPLING PROCEDURES

CAFO inspectors may be required to collect wastewater, manure, or soil samples during an inspection. Sample collection may be planned in advance or opportunistic. In addition, familiarity with sample collection is useful for determining if the facility followed appropriate procedures for sampling wastewater, manure, and soil. The facility representative may not be familiar with sampling procedures if they are collected by a consultant or extension agent.

Planned sample collection may occur when:

- EPA wants to validate results from soil and manure samples collected by the CAFO for laboratory analysis;
- Surface water or streambed sediment samples are collected as evidence to demonstrate the presence of pollutants discharged from the CAFO; or
- EPA wants to collect samples from standing water in the production area to establish pollutant concentrations.

Opportunistic sampling might occur when a facility is observed to be discharging during the inspection. Regardless, the inspector should be prepared to collect samples. Prior to the inspection, a Quality Assurance Project Plan (QAPP) should be prepared and the inspector should prepare and be familiar with sampling equipment. Sampling, analysis, preservation technique, sample holding time, and sample container requirements are provided in 40 CFR Part 136 as authorized by Section 304(h) of the CWA.

Chapter 5 of the *NPDES Compliance Inspection Manual* is a helpful reference for wastewater sampling/analysis.

Water and wastewater sampling procedures

Typically grab samples will be collected during a CAFO inspection, not composite samples. Grab samples are individual samples collected over a period of time not exceeding 15 minutes and are representative of conditions at the time the sample is collected. The collection of a grab sample is appropriate when a sample is needed to:

- Sample an effluent that does not discharge on a continuous basis
- Provide information about instantaneous concentrations of pollutants at a specific time
- Allow collection of a variable sample volume
- Corroborate composite samples

Some parameters may be sampled only by grab sampling, but others may be sampled by either grab or composite sampling. Parameters not amenable to compositing include pH, temperature, dissolved oxygen, chlorine, purgeable organics, oil and grease, coliform bacteria, and others specified in 40 CFR Part 136. Volatile organics, sulfides, phenols, and phosphorus samples can be composited but require special handling procedures. BOD and ammonia

nitrogen can be sampled by using either grab or composite techniques; if composite sampling is used, appropriate preservation must be provided during and after the sampling period.

Typical parameters sampled at CAFOs are those which readily show an effect on water quality by the discharge. These might include Biochemical Oxygen Demand (BOD), fecal or total coliform bacteria, specific conductance, and ammonia nitrogen. Many other parameters, however, may appropriately be sampled to document such discharges. Sampling of any one or a combination of these parameters can aid the inspector in documenting an illegal discharge.

The volume of samples collected depends on the type and number of analyses needed, as reflected in the parameters to be measured. Obtain the volume of the sample sufficient for all the required analyses plus an additional amount to provide for any split samples or repeat analyses. Consult the laboratory receiving the sample for any specific volume required. In addition, EPA's *Methods for Chemical Analysis of Water and Wastes* (USEPA 1979b) and *Handbook for Sampling and Sample Preservation of Water and Wastewater* (USEPA 1982), and the current Environmental Protection Agency (EPA)-approved edition of *Standard Methods for the Examination of Water and Wastewater* [American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF) contain specific recommended minimum sample volumes for different pollutant parameters.

Sample storage and holding times

40 CFR Part 136 describes required sample containers, sample preservation, and sample holding time. It is essential that the sample containers be made of chemically resistant material unaffected by the concentrations of the pollutants measured. In addition, sample containers must have a closure that will protect the sample from contamination. Collect wastewater samples for chemical analysis in plastic (polyethylene) containers. Exceptions to this general rule are organic pollutant samples which are collected in properly cleaned glass jars or bottles and sealed. Collect bacteriological samples in properly sterilized plastic or glass containers.

Ensure sample containers are clean and uncontaminated. Review analytical procedures for specific container cleaning procedures. Use precleaned and sterilized disposable containers when possible. If not, use the following procedures for cleaning sample containers:

- Wash with hot water and detergent.
- Rinse with acid (e.g., nitric for metals).
- Rinse with tap water, then rinse three or more times with organic-free water.
- Rinse glass containers with an interference-free, redistilled solvent (such as acetone or methylene chloride for extractable organics).
- Dry in contaminant-free area.

Table K 1 presents required containers, preservation techniques, and holding times for parameters that might be analyzed in a CAFO water sample.

TABLE K 1. Required containers, preservation techniques, and holding times (EPA 2004)

Parameter	Container	Preservative	Maximum Holding Time
Coliform, fecal and total	Polyethylene or glass	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
Ammonia	Polyethylene or glass	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Dissolved Oxygen Probe Winkler	Glass bottle & top Glass bottle & top	None required Fix onsite and store dark	Analyze immediately 8 hours
Chloride	Polyethylene or glass	None required	28 days
BOD ₅	Polyethylene or glass	Cool, 4°C	48 hours
Total phosphorus	Polyethylene or glass	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Nitrate	Polyethylene or glass	Cool, 4°C	48 hours

Identify each sample accurately and completely. Use labels or tags to identify the samples that are moisture-resistant and able to withstand field conditions. Use a waterproof pen to complete the labels or tags. A numbered label or tag associated with a field sample data sheet containing detailed information on the sample is preferable to using only a label or tag for information. The information for each sample should include the following:

- Facility name/location
- Sample site location
- Sample number
- Name of sample collector
- Date and time of collection
- Indication of grab or composite sample with appropriate time and volume information
- Identification of parameter to be analyzed
- Preservative used.

To ensure the validity of the permit compliance sampling data in court, written records must accurately trace the custody of each sample through all phases of the monitoring program. The primary objective of this chain-of-custody is to create an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through its analysis and introduction as evidence.

- Use sample seals to protect the sample's integrity from the time of collection to the time it is opened in the laboratory. The seal should indicate the collector's name, the date and time of sample collection, and sample identification number.

- Pack samples properly to prevent breakage. Seal or lock the shipping container to readily detect any evidence of tampering can be readily detected. Use of tamper proof evidence tape is recommended.
- Place samples on ice or synthetic ice substitute that will maintain sample temperature at 4°C throughout shipment.
- Accompany every sample with a sample tag and a chain-of-custody record that has been completed, signed, and dated. The chain-of-custody record should include the names of sample collectors, sample identification numbers, date and time of sample collection, location of sample collection, and names and signatures of all persons handling the sample in the field and in the laboratory.
- The responsibility for proper packaging, labeling, and transferring of possession of the sample lies with the inspector.
- Accompany all sample shipments with the chain-of-custody record and other pertinent forms. The originator retains a copy of these forms. Also, the originator must retain all receipts associated with the shipment.
- EPA Inspectors with the responsibility of working with hazardous materials that are placed in commerce (transporting/shipping) must have hazardous materials training as required by the Department of Transportation.
- When transferring possession of samples, the transferee must sign and record the date and time on the chain-of-custody record (use the currently approved record). In general, make custody transfers for each sample, although samples may be transferred as a group, if desired. Each person who takes custody must fill in the appropriate section of the chain-of-custody record.
- Pack and ship samples in accordance with applicable International Air Transportation Association (IATA) and/or DOT regulations.

In general, the most common monitoring errors usually are improper sampling methodology, improper preservation, and excessive sample holding time. In addition, the inspector can analyze field blanks to check for analytical artifacts and/or background introduced sampling and analytical procedures.

Field Blanks

Field blanks are distilled or de-ionized water samples prepared when you are collecting water quality samples. Field blanks are prepared, in the field, after cleaning all sampling equipment but before sample collection. Blanks are prepared by pouring distilled de-ionized water into each scoop, dipper, etc. used for sample collection and then into sample bottles as if they were actual field samples. The field blanks are processed and analyzed in an identical manner as the water quality samples. If the lab detects any contamination in the blanks, the sampling results could be considered tainted (either from contamination, errors in sampling, or analysis problems). Collection and analysis of field blanks is not required by federal CAFO regulations; however, field blanks are used for quality control to assess whether contamination was introduced during sampling, and may prove useful in interpretation of results.

Soil sampling procedures

Crop nutrient requirements vary depending on factors such as soil characteristics and previous fertilization. Soil testing is used to provide agronomic and environmentally sound nutrient and lime recommendations. It provides growers a means to assess soil pH and plant-available nutrient content, to determine the need for addition of lime and nutrients, and to minimize nutrient losses to the environment from over-application.

Good animal manure management includes routine soil sampling on every field that manure is applied. EPA generally considers soil sampling for phosphorus every 5 years as the minimum necessary to properly manage soil nutrient levels (as is required for Large dairy, beef, poultry, swine, and veal calf CAFOs under the ELG. 40 CFR Part 412.4(c)(3)).

Proper sampling is the most important component of an accurate soil test. If a representative sample is not collected, the recommendations developed by the laboratory will likely be inaccurate, resulting in excessive nutrient application or deficiencies that will affect production. Permit writers and inspectors will generally not be collecting soil samples, so this section is provided for informational purposes only. However, enforcement actions might require the soil sample collection in some cases.

A soil probe is the most efficient way to collect samples. For facilities applying nutrients at a nitrogen-based rate, collect separate soil samples at depths of 0 to 12 and 12 to 24 inches. Collect soil samples at a depth of 0 to 12 inches only at facilities applying at a phosphorus-based rate.

Every soil sample submitted for testing typically consist of about 15 to 20 cores taken at random locations throughout one field or management unit. The various cores will be used to form one composite sample to be submitted for laboratory analysis. Keep in mind that each composite sample should represent only one general soil type or condition. If the field contains areas that are obviously different in slope, color, drainage, and texture and if those areas can and will be managed separately, a separate sample should be submitted.

Manure sampling procedures

Manure is a variable material requiring proper sampling procedures to ensure collection of a representative sample. Manure samples submitted to a laboratory should represent the average composition of the material that will be applied to the field. For liquid manure, sample directly from the storage structure, from the outlet pipe where liquid is removed, or from the field using catch cans to collect samples applied through sprinklers. A minimum of six separate liquid manure subsamples must be collected. Combine the subsamples in a clean bucket, thoroughly mix, and transfer approximately one pint of liquid to a clean bottle or another rigid container.

For solid manure, remove the surface six-inch crust and use an auger or shovel to core into the pile. Take a minimum of six separate subsamples from around the pile and combine them in a clean bucket. Mix well and transfer approximately one quart to a clean plastic bag. Keep all manure samples cool until delivered to a lab.

It is important that proper containers are used and maximum holding or shipping times are also identified and followed to avoid contaminating or altering the collected samples.

Laboratory identification

The laboratory selected for analysis of surface water or discharge samples may be different from the laboratory selected for analyzing manure and soil samples. Regardless, the laboratory selected should be certified to perform the analyses according to 40 CFR Part 136 methods for water samples and state technical standards for nutrient management for soil and manure samples. Ideally, the laboratory will be able to provide sampling materials at no charge along with sample collection and preparation instructions and mailing labels (if needed).

Regulations at 40 CFR Part 123.36 requires that state technical standards for nutrient management identify acceptable labs or methods for conducting soil and manure analyses. Alternately, NRCS' CPS 590 (NRCS 2011) specifies requirements for selecting laboratories to conduct soil and manure analyses. Soil test analyses must be performed by laboratories successfully meeting the requirements and performance standards of the North American Proficiency Testing Program-Performance Assessment Program (NAPT-PAP) under the auspices of the Soil Science Society of America (SSSA) and NRCS, or other NRCS-approved program that considers laboratory performance and proficiency to assure accuracy of soil test results. Manure testing analyses must be performed by laboratories successfully meeting the requirements and performance standards of the Manure Testing Laboratory Certification program (MTLCP) under the auspices of the Minnesota Department of Agriculture, or other NRCS- approved program that considers laboratory performance and proficiency to assure accurate manure test results.

Data Handling and Reporting

Verified analytical results are normally entered into a laboratory data management system of some type. The system should contain the sampling data, including sampling time and exact location, dates and times, names of analysts, analytical methods or techniques used, and analytical results. Data are then reported to the inspector for inclusion in the compliance report.