NPDES Compliance Inspection Manual

Chapter 5

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CHAPTER 5 – SAMPLING

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A. EVALUATION OF PERMITTEE SAMPLING PROGRAM AND COMPLIANCE SAMPLING

Wastewater sampling/analysis is an integral part of the National Pollutant Discharge Elimination System (NPDES) Compliance Monitoring Program. NPDES permits contain specific and legally enforceable effluent limitations and monitoring requirements.

OBJECTIVES AND REQUIREMENTS

When evaluating the permittee sampling program, the inspector should:

- Verify that the permittee's sampling program complies with the permit.
- Verify that the permittee's sampling program complies with:
  - Title 40 of the *Code of Federal Regulations* (CFR), sections 136.1 to 136.6 and Appendices A, B, and C (Guidelines for Establishing Test Procedures for the Analysis of Pollutants) for wastewater samples; and 40 CFR Part 503.
- Document potential violations to support enforcement action.

In addition, specific objectives of the sampling conducted by inspectors include the following:

- Verify compliance with effluent limitations.
- Verify accuracy of reports and program self-monitoring.
- Support enforcement action.
- Support permit development reissuance and/or revision.
- Determine the quantity and quality of effluent.

Sampling, analysis, preservation technique, sample holding time, and sample container requirements are provided under 40 CFR Part 136 as authorized by section 304(h) of the Clean Water Act (CWA). Chapter 7 contains more information on required analytical procedures "Laboratory Analyses Techniques Evaluation." See the checklist for use in evaluating the permittee's sampling program at the end of this chapter.

For all NPDES permittees the inspector should perform a review of sampling procedures and quality control measures the facility uses to ensure the integrity of sample data.

To evaluate sampling procedures, assess the following eight areas:

- Sample site locations
- Sample collection techniques
- Field measurements
- Sample labeling (including locations) and documentation
- Sample preservation and holding time
- Transfer of custody and shipment of samples
- Quality control

- Data handling and reporting

SIGNIFICANT INDUSTRIAL USER MONITORING PROGRAM

It is the responsibility of the permitted Publicly Owned Treatment Works (POTW) with a pretreatment program to oversee sampling procedures of industrial users and to conduct compliance monitoring of its own. Therefore, during a Pretreatment Compliance Inspection (PCI) or audit, the inspector may also need to evaluate POTW sampling procedures for significant industrial users who discharge to the POTW in addition to evaluating the sampling procedures of any permitted POTW. According to the General Pretreatment Regulations, 40 CFR 403.12(o), industrial users and POTWs subject to 40 CFR 403.12 reporting requirements must maintain the following monitoring records:

- Date, exact place, method and time of sampling, and name of sampler
- Date of analysis
- Name of analyst
- Analytical techniques/methods used
- Analytical results

During a PCI or an audit, the inspector evaluates the POTW industrial user monitoring program with respect to the criteria specified in the POTW pretreatment program. Elements of the sampling scheme will include the eight areas addressed above and any other areas specifically addressed in the pretreatment program. Chapter 9 discusses the focus of this evaluation in greater detail.

BIOSOLIDS MONITORING PROGRAM

Chapter 10 discusses evaluation of a permittee's biosolids monitoring program. Lists of approved biosolids analytical methods, sample containers, preservation techniques, and holding times for biosolids samples can be found on EPA's website at: https://www.epa.gov/biosolids/additional-information-biosolids-managers#analytical.

TOXICITY TESTING PROGRAM

Chapter 8 discusses evaluation of a permittee's Whole Effluent Toxicity testing program. In addition, for methods manuals for Whole Effluent Toxicity testing go to https://www.epa.gov/cwa-methods/whole-effluent-toxicity-methods.

STORMWATER PROGRAM

Chapter 11 provides considerations for performing stormwater monitoring.

B. SAMPLING PROCEDURES AND TECHNIQUES

Whether an inspector is evaluating a permittee's sampling program or conducting compliance sampling on the permittee's effluent, that inspector must be familiar with the procedures and techniques necessary for accurate sampling of wastewaters. The following discussion details the procedures for sample collection, preservation, sample transfer including chain-of-custody, quality control, and data handling.

WASTEWATER SAMPLE COLLECTION TECHNIQUES

Sample collection is an important part of the compliance monitoring program. Without proper sample collection procedures, the results of such monitoring programs are neither useful nor valid, even with the most precise and accurate analytical measurements.

Selection of Representative Sampling Sites

Normally, samples should be collected at the location specified in the permit. In some instances, the sampling location specified in the permit may not be adequate for the collection of a representative sample. In that case, the inspector should determine the most representative sampling point available and collect a sample at that location as well as the location specified by the permit (or chosen by the permittee). If the facility disagrees, the reason for the conflict must be documented for later resolution by the permitting authority.

Sample Types

Two types of sample techniques are used: grab and composite. For many monitoring procedures, the regulations at 40 CFR Part 136 do not specify sampling type. For these procedures, the NPDES permit writer determines the appropriate sample type based on the data objective, and/or the required analytical method and specifies the sampling technique in the NPDES permit.

Grab Samples. Grab samples are individual samples collected at a specific time not exceeding 15 minutes and are representative of the conditions at the time the sample is collected. The sample volume depends on the type and number of analyses to be performed. The collection of a grab sample is appropriate when a sample is needed to:

- Represent 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.
- Monitor parameters not amenable to compositing (e.g., pH, temperature, dissolved oxygen, chlorine, purgeable organics, oil and grease, coliform bacteria, and others specified by the NPDES permit, which may include phenols, sulfites, and hexavalent chromium).

Composite Samples. Composite samples are samples collected over time, either by continuous sampling or by mixing discrete samples. Composite samples represent the average characteristics of the wastestream during the compositing period. Composite samples are collected when:

- Average pollutant concentration during the compositing period is desired.
- Mass per unit time loadings are calculated.

- Wastewater characteristics are highly variable.

The four primary methods of composite sample collection are time compositing, flow proportion compositing, sequential compositing, and continuous compositing. Table 5-1 lists the advantages and disadvantages of these methods. The permit may specify which type of composite sample to use. Composite samples are collected either manually by combining multiple grab samples or by using automatic sampling equipment. Inspectors should consider variability in wastestream flow rate, parameter concentrations and the approved EPA methods when choosing compositing methods, sampling equipment (tubing and containers), and quality assurance procedures. The compositing methods are as follows:

- Time Composite Sample: This method requires discrete sample aliquots collected in one container at constant time intervals. This method is appropriate when the flow of the sampled stream is constant (flow rate does not vary more than ±10 percent of the average flow rate) or when flow monitoring equipment is not available.

Table 5-1. Compositing Methods

Method	Advantages	Disadvantages	Comments
Time Composite			
Constant sample volume, constant time interval between samples.	Minimal manual effort; requires no flow measurement.	May lack representativeness for highly variable flows.	Widely used in both automatic and manual sampling.
Flow-Proportional Composite			
Constant sample volume, time interval between samples proportional to stream flow.	Minimal manual effort.	Requires accurate flow measurement reading equipment; manual compositing from flowchart.	Widely used in automatic as well as manual sampling.
Constant time interval between samples, sample volume proportional to total stream flow at time of sampling.	Minimal instrumentation.	Manual compositing from flowchart in absence of prior information on the ratio of minimum to maximum flow; chance of collecting too small or too large individual discrete samples for a given composite volume.	Used in automatic samplers and widely used as manual method.
Constant time interval between samples, sample volume proportional to total stream flow since last sample.	Minimal instrumentation.	Manual compositing from flow chart in absence of prior information on the ratio of minimum to maximum flow; chance of collecting too small or too large individual discrete samples for a given composite volume.	Not widely used in automatic samplers but may be done manually.

Table 5-1. Compositing Methods

Method	Advantages	Disadvantages	Comments
Sequential Composite			
Series of short period composites, constant time intervals between samples.	Useful if fluctuations occur and the time history is desired.	Requires manual compositing of aliquots based on flow.	Commonly used; however, manual compositing is labor intensive.
Series of short period composites, aliquots taken at constant discharge increments.	Useful if fluctuations occur and the time history is desired.	Requires flow totalizer; requires manual compositing of aliquots based on flow.	Manual compositing is labor intensive.
Continuous Composite			
Constant sample volume.	Minimal manual effort, requires no flow measurement highly variable flows.	Requires large sample capacity; may lack representativeness for highly variable flows.	Practical but not widely used.
Sample volume proportional to stream flow.	Minimal manual effort, most representative especially for highly variable sample volume, variable pumping capacity and power.	Requires accurate flow measurement equipment, large sample volume, variable pumping capacity, and power.	Not widely used.

- Flow-Proportional Composite Sample—in one method, a constant sample volume is collected at varying time intervals proportional to stream flow (e.g., 200 milliliters sample collected for every 5,000 gallons of flow). In the other method (which has two variations, see Table 5-1), the sample is collected by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots.
- Sequential Composite Sample—this method requires discrete samples collected in individual containers at constant time intervals or discharge increments; for example, samples collected every 15 minutes, composited into separate containers each hour. The discrete samples can then be manually flow-proportioned to form the composite sample. Alternatively, a constant sample volume is collected at constant discharge volume increments measured with a flow totalizer.
- Continuous Composite Sample—collect this sample continuously from the wastestream. The sample may be constant volume, or the volume may vary in proportion to the flow rate of the wastestream.

Influent Sample Collection. Document and take influent samples at points of high turbulence flow to ensure good mixing. In some instances, the most desirable location may not be accessible. Ensure sampling points are located prior to any internal facility return lines, and sampling equipment should be placed so that it does not interfere with flow measuring devices. The preferred sampling points for raw wastewater are at the most downstream location from the collection lines, but prior to preliminary treatment:

- Waste flowing from the last process in a manufacturing operation, for an industrial user.
- Pump wet well (if turbulent).
- Upstream collection lines, tank, or distribution box following pumping from the wet well or sump.
- Flume throat.
- Aerated grit chamber.
- Upstream siphon following the comminutor (in absence of grit chamber).

If it is not possible to sample at a preferred point, choose an alternative location and document the basis for choosing that location.

Effluent Sample Collection. Collect effluent samples at the location specified in the NPDES permit. Occasionally, municipal plant permits may specify sampling prior to chlorination. For these plants, monitor all parameters at the upstream location except fecal coliforms, pH, and total residual chlorine. Collect wastewater for use in bioassays at the location specified in the facility's NPDES permit.

Collect samples either manually (grab or composite) or with automatic samplers (continuous or composite). The following general guidelines apply when taking samples:

- Take samples at a location specified in the NPDES permit and/or at a location selected to yield a representative sample.
- Use the sampling method (grab, composite, continuous) specified in the permit. Some parameters that must be collected as an individual grab sample are dissolved oxygen, total residual chlorine, oil and grease, coliform bacteria, purgeable organics, sulfides, cyanide, and total phenols.
- Avoid collecting large nonhomogeneous particles and objects.
- Collect the sample facing upstream to avoid contamination.
- Do not rinse sample container with sample when collecting oil and grease and microbiological samples, but fill the container directly to within 2.5 to 5 cm from the top.
- Fill the container completely if the sample is to be analyzed for purgeable organics, oxygen, ammonia, hydrogen sulfide, free chlorine, pH, hardness, sulfite, ammonium, ferrous iron, acidity, or alkalinity.
- Collect sufficient volume to allow for quality assurance testing. (see EPA’s website https://www.epa.gov/cwa-methods for a listing of all approved sampling methods. Each sampling method will indicate the required sampling equipment, sampling containers and sampling volume, but additional volumes may be necessary for quality assurance testing.)

The following general guidelines apply when using automatic samplers:

- Collect samples where the wastewater is well mixed. Collect the sample near the center of the flow channel at 0.4 to 0.6 depth (mid-depth).
- Obtain a sufficient volume of sample to perform all required analyses plus any additional amount for quality control. Individual portions of a composite sample should be at least 100 milliliters to minimize sampler solids bias.
- For automatic samplers that use a peristaltic pump, obtain adequate flow rates in the sampler tubing to effectively transport the suspended solids. To avoid solids bias, the velocity of the wastewater in sample tubing should be at least 2 feet per second (fps) and the tubing diameter should be at least 0.25 inch.
- Time of sample collection begins when the last aliquot is dispensed into the composite sample container.

Sample Volume

The volume of sample 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. EPA approved sampling methods provide a guide to sample volumes required for determining the constituents in wastewater (available at https://www.epa.gov/cwa-methods). Consult the laboratory receiving the sample for any specific volume required. EPA's *Methods for Chemical Analysis of Water and Wastes* (EPA, 1979a) and *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA, 1982), and the current 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), 2013) contain specific recommended minimum sample volumes for different pollutant parameters.

Sample Containers

The regulations at 40 CFR Part 136 describe required sample containers, sample preservation, and sample holding time. EPA approved sampling methods indicate appropriate sample containers for each analysis 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 oil and grease samples, pesticides, phenols, polychlorinated biphenyls (PCBs), and other organic pollutant samples. Collect these in properly cleaned glass jars or bottles and seal. Collect bacteriological samples in properly sterilized plastic or glass containers. Collect samples that contain constituents that will oxidize when exposed to sunlight (such as iron cyanide complexes) in dark containers.

Ensure sample containers are clean and uncontaminated. Check analytical procedures to determine if they specify container cleaning procedures. Use precleaned and sterilized disposable containers (e.g., polyethylene cubitainers). If these are not used or if the analytical method does not specify procedures, 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.

EPA SAMPLE IDENTIFICATION METHODS

Identify each sample accurately and completely. Use labels or tags to identify the samples that are moisture-resistant and able to withstand field conditions. If moisture-resistant labels are not available, place a piece of tape over each label to prevent water damage. 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[2]. 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
- If the sample is preserved and, if so, the preservative used

WASTEWATER SAMPLE PRESERVATION AND HOLDING TIME

In most cases, wastewater samples contain one or more unstable pollutants that require immediate (e.g., within 15 minutes) preservation and/or analysis. Provide appropriate chemical preservation before transferring samples to the laboratory. EPA approved sampling methods indicate appropriate sample preservation for each analysis (sampling methods are available at https://www.epa.gov/cwa-methods). Procedures used to preserve samples include cooling, pH adjustment, and chemical treatment. For some parameters, such as cyanide and phenols, add preservatives to sample bottles prior to or immediately following sample collection. For many samples, if preservatives are not appropriately used, bacteria can quickly degrade certain constituents (such as phenols and phosphorus). Other constituents may volatilize (such as volatile organics and sulfides) or may react to form different chemical species (hexavalent

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[2] Note: Preprinted labels, data sheets, chain-of-custody forms, etc., can be done in the field using software developed by the Superfund Program.

chromium, for example). Proper preservation and holding times are essential to ensure sample integrity (see 40 CFR Part 136).

Analysis of samples within one day ensures against error from sample deterioration. However, such prompt analysis is not feasible for composite samples in which portions may be stored for as long as 24 hours. Where possible, provide sample preservation during compositing, usually by refrigeration to 6°C (or icing). If using an automatic sampler with ice, replace the ice as necessary to maintain low temperatures. This is a limitation of automatic samplers used during the summer when ice must be frequently replaced.

Table II of 40 CFR 136.3(e) indicates maximum sample holding times. Times listed are the maximum holding times between sample collection and analysis that are allowed for the sample to be considered valid. Unless otherwise specified in the method, holding time limitations begin upon combination of the last aliquot in a sample. When use of an automatic sampler makes it impossible to preserve each aliquot, the chemical samples may be preserved by maintaining at 6°C until compositing and sample splitting is completed (40 CFR 136.3(e)).

TRANSFER OF CUSTODY AND SHIPMENT OF SAMPLES

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 (EPA Order 5360.1). The primary objective of this chain-of-custody is to create an accurate written record (see an example chain-of-custody form in Appendix M) 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. The following procedures are appropriate for the transfer of custody and shipment of samples:

- Use sample seals to protect the sample's integrity from the time of collection to the time it is opened in the laboratory, including the time the sample is within an automatic sampling apparatus, thus the automatic sampler should be sealed on the outside. The seal should indicate the collector's name, the date and time of sample collection, and sample identification number. For automatic samplers, seals should indicate the sample time at which the apparatus began sampling, as the sample container is subsequently sealed in the apparatus.
- Pack samples properly to prevent breakage. Seal or lock the shipping container to readily detect any evidence of tampering. Use of tamper-proof evidence tape is recommended.
- Place samples on ice or synthetic ice substitute that will maintain sample temperature at 6°C throughout shipment.
- The responsibility for proper packaging, labeling, and transferring of possession of the sample lies with the inspector. 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 originator retains a copy of the chain of custody 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 (see Appendix N).
- 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, custody transfers are made for each sample, although samples may be transferred as a group, if desired. For each sample being transferred, the transferee should list the sample and their name on the custody record. Each person who takes custody must fill in the appropriate section of the chain-of-custody record. Both the transferee and person who takes custody of the sample(s) must sign the custody record.
- Pack and ship samples in accordance with applicable International Air Transportation Association (IATA) and/or DOT regulations.

QUALITY CONTROL

Conduct control checks during the actual sample collection to determine the performance of sample collection techniques. In general, the most common monitoring errors usually are improper sampling methodology, improper preservation, inadequate mixing during compositing and splitting, and excessive sample holding time. In addition, collect and analyze the following samples to check sample collection techniques:

Blanks

- Trip blank. Trip blanks are vial(s) filled at the laboratory with deionized water. The blank(s) follows the same handling and transport procedures as the samples collected during the event. The blank(s) functions as a check on sample contamination originating from sample transport, shipping and from site conditions.

  Note: Expose the trip blank vial(s), to the same environmental conditions (light, temperature, etc.) of the sample vial(s) but do not open until it is time for analysis.

- Field blank/field reagent blank. Field blanks are similar to trip blanks except they are prepared in the field with deionized water exactly as the sample(s) that are collected. Field blanks are used to check for analytical artifacts and/or background introduced by sampling and analytical procedures.
- Temperature blank. A temperature blank is a small sample bottle filled with distilled water that is placed in each cooler prior to shipment. Upon arrival at the laboratory the temperature of the sample bottle is measured to evaluate if samples were adequately cooled during sample shipment.
- Equipment/rinsate blank. Collect an equipment/rinsate blank when using an automatic sampler or other non-dedicated equipment during the sampling process. The blank is a check of the equipment cleanliness. For automatic samplers, prepare blanks prior to collecting samples, by pumping deionized organic free water (rinsate) through the

sampler and collecting the discharge purge water in a sample container for analysis for the constituents of concern.

Field Duplicate. Collect a field duplicate sample simultaneously from the same source at selected stations on a random timeframe by grab samples or from two sets of field equipment installed at the site. Duplicate samples check analytical precision as well as evaluate the "representativeness" of the sample aliquot.

Split Samples. Split samples are samples that have been divided into two containers for analysis by separate laboratories. These samples provide an excellent means of identifying discrepancies in the permittee's analytical techniques and procedures. When filling split samples from a single composite jug, shake the composited sample well and half fill the EPA sample container, then shake the composite again and fill half of the permittee's container. Repeat the procedure for each parameter collected.

The laboratories performing the sample analyses should also use the following control measures:

Prep/Reagent Blank. A prep/reagent blank is a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and to aid in identifying errors in the observed value that may result from the analytical steps.

Quality Control Sample. A quality control sample is an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. Use this sample to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurements' system.

Matrix Spike/Matrix Spike Duplicate (MS/MSD). A matrix spike/matrix spike duplicate sample is three times the normal volume required for a specific chemical analysis to which a known quantity of analyte has been added prior to all sample preparation. The laboratory utilizes the MS/MSD samples as part of their Quality Assurance/Quality Control Program.

- Use a matrix spike to verify accuracy of the analytical procedures.
- A matrix spike duplicate is a duplicate of a matrix spike sample. It measures the precision of the analysis in terms of relative percent difference.

Table 5-2 indicates quality control procedures for field analyses and equipment. Quality control is discussed in greater detail in Chapter 7 of this manual and EPA's *NPDES Compliance Inspector Training module: Laboratory Analyses* (EPA, 1990).

Table 5-2. Quality Control Procedures for Field Analysis and Equipment

Parameter	General	Daily	Other Frequency
Dissolved Oxygen			
Membrane Electrode	• Enter the make, model, and serial and/or ID number for each meter in a logbook. • Report data to nearest 0.1 mg/L.	• Calibrate meter using manufacturer's instructions or Winkler-Azide method. • Check membrane for air bubbles and holes. Change membrane and Potassium chloride (KCl) solution if necessary. • Check leads, switch contacts, etc., for corrosion and shorts if meter pointer remains off-scale.	• Annually, check instrument calibration and linearity using a series of at least three dissolved oxygen standards. • Annually, take all meters to the laboratory for maintenance, calibration, and quality control checks.
Winkler-Azide Method	Record data to nearest 0.1 mg/L.	Duplicate analysis should be run as a precision check. Duplicate values should agree within ±0.2 mg/l.	
pH			
Electrode Method	Enter the make, model, and serial and/or ID number for each meter in a logbook.	• Calibrate the system against traceable standard buffer solutions of known pH value that closely brackets the actual sample pH (e.g., 4, 7, and 10 at the start of a sampling run). • Periodically check the buffers during the sample run and record the data in the logbook. • Be on the alert for erratic meter response arising from weak batteries, cracked electrodes, fouling, etc. • Check response and linearity following highly acidic or alkaline samples. Allow additional time for equilibration. • Check against the closest reference solution each time a violation is found. • Rinse electrodes thoroughly between samples and after calibration. Blot dry. • Store the probe in approved storage solution (e.g., KCl)	

Table 5-2. Quality Control Procedures for Field Analysis and Equipment

Parameter	General	Daily	Other Frequency
Conductivity			
	Enter the make, model, and serial and/or ID number for each meter in a logbook.	• Standardize with KCl standard solutions having similar specific conductance values to those anticipated in the samples. Calculate the cell constant using two different standards. • Rinse cell after each sample to prevent carryover.	• Quarterly, take all meters to lab for maintenance, calibration, and quality control checks. • Quarterly, check temperature compensation. • Quarterly, check date of last platinizing, if necessary. • Quarterly, analyze NIST or EPA reference standard solutions, and record actual vs. observed readings in the logbook.
Residual Chlorine			
Amperometric Titration	Enter the make, model, and ID and/or serial number of each titration apparatus in a logbook. Report results to nearest 0.01 mg/l.	Refer to instrument manufacturer's instructions for proper operation and calibration procedures.	Biweekly, return instrument to lab for maintenance and addition of fresh, standardized reagents.
Temperature			
Manual Thermometer	• Enter the make, model, and serial and/or ID number and temperature range. • All standardization should be against a traceable NIST or NIST calibrated thermometer. Reading should agree within ±1°C. If enforcement action is anticipated, calibrate the thermometer before and after analysis. All data should be read to the nearest 1°C. Report data between 10° and 99°C to two significant figures.	Check for air spaces of bubbles in the column, cracks, etc. Compare with a known source if available.	• Initially and annually, determine accuracy throughout the expected working range of 0°C to 50°C. A minimum of three temperatures within the range should be used to verify accuracy. Preferably, the 3 temperature readings should be taken within the following ranges: 5–10°C, 15–25°C, and 35–45°C.
Thermistors, Thermographs	Enter the make, model, and serial and/or ID number of the instrument in a logbook. All standardization shall be against a NIST or NIST calibrated thermometer. Reading	Check thermistor and sensing device for response and operation according to the manufacturer's instruction. Record actual versus standard temperature in logbook.	Initially and annually, determine accuracy throughout the expected working range of 0°C to 50°C. A minimum of three temperatures within the range should be used to verify

Table 5-2. Quality Control Procedures for Field Analysis and Equipment

Parameter	General	Daily	Other Frequency
	should agree within ±1°C. If enforcement action is anticipated, refer to the procedure listed above.		accuracy. Preferably, the 3 temperature readings should be taken within the following ranges: 5–10°C, 15–25°C, and 35–45°C.
Flow Measurement			
	Enter the make, model, and serial and/or ID number of each flow measurement instrument in a logbook.	Install the device in accordance with the manufacturer's instructions and with the procedures given in owner's manual.	Annually affix record of calibration (as per NIST standard or manufacturer's suggested standard) to the instrument log.
Automatic Samplers			
	Enter the make, model, and serial and/or ID number of each sampler in a logbook.		For each sampling event, check intake velocity vs. head (using a minimum of three samples), and clock time setting vs. actual time interval. Calibrate annually and record results in a logbook.

QUALITY ASSURANCE PROJECT PLAN

The EPA has developed the Quality Assurance Project Plan (QAPP) as a tool for project managers and planners to document the type and quality of data needed for the agency to make environmental decisions and to describe the methods for collecting and assessing those data. The QAPP is required for all EPA projects resulting in the generation, collection, and use of environmental data. The development, review, approval and implementation of the QAPP is an integral part of an Agency-wide Quality System, which is required per the authority of EPA Order 5360.1 A2.

If the EPA is to have confidence in the quality of data used to support environmental decisions, there must be a systematic planning process in place. A product of the systematic planning process is the QAPP. An example of the systematic planning process endorsed by the EPA is the Data Quality Objectives (DQO) Process. The QAPP ensures that the needed management and technical practices are in place so that environmental data used to support agency decisions are of adequate quality and usability for their intended purpose.

Prior to the start of data collection, a QAPP defining the goals and scope of the project, the need for sample collection, a description of the data quality objectives and QA/QC activities to ensure data validity and usability must be developed by the project officer. Thereafter, a review by all parties to the sampling effort, such as a Quality Assurance (QA) Officer, must be conducted. Also, EPA laboratories will require a copy of an approved QAPP prior to conducting any sample analysis. This QAPP requirement applies to both EPA staff and outside contractors. The process for approval of the QAPP and other documents related to the data collection activity should be outlined in the lead organization's Quality Management Plan (QMP).

For further information on QAPP's please visit the Office of Environmental Information (OEI) web page at: https://www.epa.gov/quality/agency-wide-quality-system-documents.

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 time and exact location, analysis dates and times, names of analysts, analytical methods/techniques used, and analytical results. Data are then reported to the inspector for inclusion into the compliance report. The quality assurance manual by EPA (EPA, 1979b) and the article by J.J. Delfino (Delfino, 1977) provide useful information to the inspector on many data management techniques.

C. REFERENCES

The following is a list of resources providing additional information on sampling.

American Public Health Association (APHA), American Water Works Association (AWWA), and World Economic Forum (WEF). (2013). *Standard Methods for the Examination of Water and Wastewater*.

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D. PERMITTEE SAMPLING INSPECTION CHECKLIST

A. PERMITTEE SAMPLING EVALUATION			
Yes	No	N/A	1. Take samples at sites specified in permit.
Yes	No	N/A	2. Locations adequate for representative samples.
Yes	No	N/A	3. Flow proportioned samples obtained when required by permit.
Yes	No	N/A	4. Complete sampling and analysis on parameters specified by permit.
Yes	No	N/A	5. Conduct sampling and analysis in frequency specified by permit.
Yes	No	N/A	6. Permittee uses method of sample collection required by permit. Required method: _______________________________________ If not, method being used is: ( ) Grab ( ) Manual Composite ( ) Automatic Composite
			7. Sample collection procedures adequate:
Yes	No	N/A	a. Samples refrigerated during compositing.
Yes	No	N/A	b. Proper preservation techniques used.
Yes	No	N/A	c. Containers and sample holding times before analyses conform to 40 CFR 136.3.
Yes	No	N/A	d. Samples analyzed in timeframe needed.
Yes	No	N/A	8. Facility performs monitoring and analyses more often than required by permit; if so, results reported in permittee's self-monitoring report.
Yes	No	N/A	9. Samples contain chlorine.
Yes	No	N/A	10. Use contract laboratory for sample analysis.
Yes	No	N/A	11. POTW collects samples from industrial users in pretreatment program.
B. SAMPLING INSPECTION PROCEDURES AND OBSERVATIONS			
Yes	No	N/A	1. Obtain grab samples.
Yes	No	N/A	2. Obtain composite sample. Compositing Frequency: __________________ Preservation: ________________
Yes	No	N/A	3. Refrigerate sample during compositing.
Yes	No	N/A	4. Obtain flow-proportioned sample.
Yes	No	N/A	5. Obtain sample from facility sampling device.
Yes	No	N/A	6. Sample representative of volume and nature of discharge.
Yes	No	N/A	7. Sample split with permittee.
Yes	No	N/A	8. Employ chain-of-custody procedures.
Yes	No	N/A	9. Samples collected in accordance with permit.
Yes	No	N/A	10. Observe excessive foam, grease, floating solids at the outfall.

C. AUTOMATIC SAMPLER PROCEDURES AND OBSERVATIONS			
Yes	No	N/A	1. Sample intake tubing place in a well-mixed, representative location (0.4 to 0.6 depth).
Yes	No	N/A	2. Individual aliquot volume checked and at least 100ml.
Yes	No	N/A	3. Proper sample tubing (Teflon™ for organics, otherwise Tygon®) and tubing at ID at least 0.25 inch.
Yes	No	N/A	4. Proper composite sample container (glass for organics, otherwise plastic.
Yes	No	N/A	5. Proper refrigeration (6°C or ice), with required documentation.
Yes	No	N/A	6. Proper wastewater velocity in the sample tubing (at least 2 fps).