NPDES Compliance Inspection Manual

Chapter 7



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CHAPTER 7 – LABORATORY PROCEDURES AND QUALITY ASSURANCE

Contents

| Α. | Objectives and Requirements | 134 |
|----|---|-----|
| В. | Sample Handling Procedures | 134 |
| | Evaluation of Permittee Sample Handling Procedures | 134 |
| С. | Laboratory Analyses Techniques Evaluation | 135 |
| | Evaluation of Permittee Laboratory Analytical Procedures | 135 |
| | Evaluation of Permittee Laboratory Facilities and Equipment | 136 |
| D. | Quality Assurance and Quality Control | 138 |
| | Evaluation of the Precision and Accuracy of the Permittee Laboratory | 138 |
| | Example of Laboratory QA/QC Measures for Microbial Analyses | 140 |
| | Evaluation of Permittee Data Handling and Reporting | 140 |
| | Evaluation of Permittee Laboratory Personnel | 141 |
| | Evaluation of Contract Laboratories | 141 |
| | Overview of the Discharge Monitoring Report Quality Assurance Program and How | |
| | It Relates to the Inspection Program | 141 |
| Ε. | References | 142 |
| F. | Laboratory Quality Assurance Checklist | 144 |

A. OBJECTIVES AND REQUIREMENTS

The analytical laboratory provides both qualitative and quantitative information for determining the extent of permittee compliance with permit discharge requirements. To be valuable or useful, the data must be representative and accurately describe the characteristics and concentrations of constituents in the samples submitted to the laboratory. The objectives of laboratory Quality Assurance (QA) are to monitor and document the accuracy and precision of the results reported and to meet reliability requirements.

QA refers to a total program for ensuring the reliability of data by utilizing administrative and technical procedures and policies regarding personnel, resources, and facilities. QA is required for all functions bearing on environmental measurements and includes activities such as project/study definition; sample collection and tracking; laboratory analysis; data validation, analysis, reduction, and reporting; documentation; and data storage systems. Thus, the QA program is designed to evaluate and maintain the desired quality of data. Quality Control (QC), a function of QA, is the routine application of procedures for controlling the accuracy and precision of the measurement process and includes the proper calibration of instruments and the use of the appropriate analytical procedures.

The regulations at Title 40 of the *Code of Federal Regulations* (CFR), section 122.41(e) (conditions applicable to all permits), requires adequate laboratory and process controls, including appropriate QA/QC procedures. Each permittee's laboratory must have a QA/QC program. The laboratory must document the QA/QC program in a written QA/QC manual and the laboratory should make it available to all personnel responsible for sample analyses. The manual must clearly identify the individuals involved in the QA program and document their responsibilities. The laboratory's standard operating procedures must meet user requirements in terms of specificity, completeness, precision, accuracy, representativeness, and comparability of the required testing procedures. The laboratory should devote approximately 10 to 20 percent of their resources to their QA/QC program.

Guidance in this chapter is broad-based and may not be applicable to every laboratory. This chapter includes a Laboratory Quality Assurance Checklist for the inspector's use at the end of the chapter. For detailed information concerning laboratory QA/QC, refer to Environmental Protection Agency's (EPA's) *Handbook for Analytical Quality Control in Water and Wastewater Laboratories* (EPA, 1979) and EPA's *National Pollutant Discharge Elimination System (NPDES) Compliance Monitoring Inspector Training Module: Laboratory Analysis* (EPA, 1990). If a more detailed assessment of a laboratory is required, personnel with more extensive knowledge of the methodologies should perform the inspection.

B. SAMPLE HANDLING PROCEDURES

EVALUATION OF PERMITTEE SAMPLE HANDLING PROCEDURES

Proper sample handling procedures are necessary in the laboratory from the sample's receipt to its discard. Sample handling procedures for small permittees may differ from procedures for larger permittees because staff organizational structures and treatment facility designs vary

from one facility to the next. However, proper sample handling procedures should be standardized, utilized and documented by all permittees. In evaluating laboratory sample handling procedures, the inspector should verify the following:

- The laboratory area is secure and restricts entry to authorized personnel only.
- The laboratory has a sample security area that is dry, clean, and isolated; has sufficient refrigerated space; and can be locked securely.
- The laboratory has a sample custodian and a back-up custodian.
- The custodian receives all incoming samples, signs the chain-of-custody record sheet accompanying the samples, and locks the samples in the sample security area refrigerator.
- The custodian ensures that samples are properly stored.
- The custodian performs or analyzes checks of proper preservation, container type, and holding times and documents the results.
- The custodian distributes and retrieves samples to and from personnel who perform the analyses (i.e., analysts) and documents the transfer of the samples in the chain-of-custody record, which is retained as a permanent record. The chain-of-custody record typically identifies the sample identification number, sample collection date and time, sample type, sample location, sample volume, and preservatives.
- The custodian and analysts ensure the minimum possible number of people handle the samples.
- The custodian only disposes of samples and records upon direction from the laboratory director, in consultation with previously designated enforcement officials, when it is certain that the information is no longer required or that the samples have deteriorated.

C. LABORATORY ANALYSES TECHNIQUES EVALUATION

EVALUATION OF PERMITTEE LABORATORY ANALYTICAL PROCEDURES

The permittee's laboratories or its contract laboratories must use uniform methods, thus, eliminating methodology as a variable when data are compared or shared among laboratories. The permittee's laboratory must consult 40 CFR Part 136 for the alternative methods approval process. A permittee may only use alternative test procedures if the procedures have EPA approval, as specified by 40 CFR 136.4 and 136.5, and promulgated under Public Law (PL) 92-500.

Many standardized test procedures promulgated under 40 CFR Part 136 are covered in EPA's *Methods for Chemical Analysis of Water and Wastes* (EPA, 1983) and the latest accepted 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). Revisions and new additions to this manual are made whenever new analytical techniques or instruments are developed. These are considered

accepted after final publication in the Federal Register.³ Other approved methods from United States Geological Survey (USGS), American Society for Testing and Materials (ASTM), and several commercial vendor methods are also referenced in 40 CFR Part 136.

In evaluating laboratory analytical procedures, the inspector should verify the following:

- The laboratory personnel follow analytical methods specified in the most current 40 CFR Part 136.
- The laboratory personnel properly perform any deviations allowed by 40 CFR Part 136 and maintain documentation of any EPA-approved deviation from specified procedures.
- The laboratory personnel follow QA/QC procedures that conform to the procedures specified in the permit, analytical method, or methods compendium for approved 40 CFR Part 136 methods from a consensus organization. For example, the Standard Methods for the Examination of Water and Wastewater (APHA, AWWA, and WEF) contains QA/QC procedures.
- The laboratory personnel maintain a QA/QC record on reagent preparation, instrument calibration and maintenance, incubator temperature, and purchase of supplies.
- The laboratory personnel conduct QA/QC checks on materials, supplies, equipment, instrument calibration and maintenance, facilities, analyses, and standard solutions.

EVALUATION OF PERMITTEE LABORATORY FACILITIES AND EQUIPMENT

To verify that the proper analytical procedures are being followed, the inspector should have the responsible analyst describe each of the procedures. The inspector should be alert to any deviation from the specified analytical method. Any questions regarding the proper procedures can be resolved by referring to the cited methodology. Even simple analyses can yield invalid results if the methodology cited in 40 CFR Part 136 is not exactly followed. Certain required deviations from the approved methods are cited in 40 CFR Part 136, notes.

Laboratory Services

The availability of laboratory services affects data reliability. In evaluating laboratory services, the inspector should verify that the laboratory provides the following:

- Adequate supply of laboratory pure water, free from chemical interferences and other undesirable contaminants. The laboratory personnel should check water quality routinely and document it.
- Adequate bench, instrumentation, storage, and recordkeeping space.
- Clean and orderly work area to help avoid contamination.
- Adequate circulation and egress.
- Adequate humidity and temperature control.
- Adequate lighting and ventilation.

³ The most current 40 CFR Part 136 may supersede any method or technique cited in this manual.

- Dry, uncontaminated compressed air when required.
- Efficient fume hood systems.
- Necessary equipment such as a hot plate, incubator, water bath, refrigerator for samples, glassware, pH meter, thermometer, balance, etc.
- Electrical power for routine laboratory use and, if appropriate, voltage-regulated sources for delicate electronic instruments.
- Vibration-free area for accurate weighing.

The inspector should also check that the laboratory personnel use proper safety equipment (e.g., lab coats, gloves, safety glasses, goggles, and fume hoods) where necessary. The inspector should document any problems and refer to the proper authority (e.g., Occupational Safety and Health Administration (OSHA)).

Instruments and Equipment

Instrumentation is extremely important in the analytical laboratory. To a certain extent, analytical instrumentation is always developmental; manufacturers are continually redesigning and upgrading their products, striving for miniaturization, enhanced durability and sensitivity, and improved automation. In evaluating laboratory instruments and equipment, the inspector should verify the following:

- The laboratory personnel follow standard and specific procedures for selecting and cleaning glassware and containers. Chapter 2 of EPA's *NPDES Compliance Monitoring Inspector Training Module: Laboratory Analysis* (EPA, 1990) contains detailed information on glassware cleaning.
- The laboratory personnel follow written requirements (e.g., standard operating procedures) for daily operation of instruments and equipment.
- The laboratory contains emergency equipment such as a fire extinguisher, eye wash station, shower, first aid kit, lab coats, gloves, and goggles.
- Standards and appropriate blanks are available from suppliers to perform standard calibration procedures. The laboratory personnel should use standard concentrations that closely bracket actual sample concentrations. Sources of standards are documented and where possible, traceable to a national standard (e.g., National Institute of Standards and Technology (NIST).
- The laboratory personnel maintain records of each set of analyses performed including the order in which calibration, QA/QC, and samples were analyzed (i.e., analysis run logs or instrument run logs).
- The laboratory personnel follow written troubleshooting procedures to identify common equipment malfunctions.
- The laboratory personnel follow written schedules for replacement, cleaning, checking, and/or adjustment by service personnel.
- The laboratory personnel maintain documentation on equipment maintenance and service checks.

Commonly used analytical instruments include analytical balances, pH meters, dissolved oxygen meters, conductivity meters, turbidity meters, spectrophotometers, atomic absorption spectrophotometers, organic carbon analyzers, selective ion analyzers, gas-liquid chromatographs, titrimetric analyses, and temperature controls. Chapter 2 of EPA's *NPDES Compliance Monitoring Inspector Training Module: Laboratory Analysis* (EPA 1990) includes a detailed discussion on these instruments.

Supplies

Chemical reagents, solvents, and gases are available in many grades of purity, ranging from technical grade to various ultrapure grades. The purity of the materials required in analytical chemistry varies with the type of analysis. The parameter being measured, the analytical method, and the sensitivity and specificity of the detection system determine the purity of the reagents required. Do not use reagents of lesser purity than that specified by the method. In evaluating laboratory supplies, the inspector should verify that the laboratory personnel:

- Check the accuracy of purchased solutions as per method requirements.
- Prepare stock solutions and standards using volumetric glassware.
- Prepare and standardize reagents against reliable primary standards.
- Use the required reagent purity for the specific analytical method.
- Check working standards frequently to determine changes in concentration or composition.
- Verify concentrations of stock solutions before being used to prepare new working standards.
- Label standards and reagents properly including the preparation date, concentration, the analyst's identification, storage requirements, and discard date.
- Store standards, reagents, and solvents in appropriate containers and under required method conditions and manufacturer's directions. If conditions are not specified, store standards and reagents according to 40 CFR Part 136, Table II.
- Store standards, reagents and solvents using clean containers of suitable composition with tight-fitting stoppers.
- Discard standards and reagents after recommended shelf-life has expired or when signs of discoloration, formation of precipitates, or significant changes in concentrations are observed.

D. QUALITY ASSURANCE AND QUALITY CONTROL

EVALUATION OF THE PRECISION AND ACCURACY OF THE PERMITTEE LABORATORY

The purpose of laboratory control procedures is to ensure high-quality analyses using control samples, control charts, reference materials, and instrument calibration. The laboratory must initiate and maintain controls throughout the analysis of samples. Specifically, each testing batch must contain at least one blank, standard, duplicate, and spiked (as applicable) sample analysis. When a batch contains more than 10 samples, every tenth sample should be followed by a duplicate and a spike (as applicable). Consult each method for specific QC requirements.

The precision of laboratory findings refers to the reproducibility or degree of agreement among replicate measurements of the same quantity. The closer the numerical values of the measurements come to each other, the more precise the measurements are. In a laboratory QC program, precision is determined by the analysis of actual samples in duplicate. These may represent a range of concentrations and a variety of interfering materials usually encountered during the analysis. Accuracy refers to the degree of difference between observed values and known or actual values. The closer the value of the measurement comes to the actual value, the more accurate the measurement is. The accuracy of a method can be determined by analyses of samples to which known amounts of reference standards have been added (spiked samples).

In evaluating the precision of the measurement process, the inspector should verify that the laboratory personnel:

- Introduce duplicate samples into the train of actual samples at least 10 percent of the time to monitor the performance of the analytical system.
- Prepare and use precision control charts or other statistical techniques for each analytical procedure. Develop precision control charts by collecting data from a minimum of 15 to 20 duplicate samples (run in controlled conditions) over an extended period (e.g., 10 to 20 days). Statistical methods include calculation of mean, standard deviation, and variance to define the range and variability of the data.
- Take corrective actions when data fall outside the warning and control limits.
- Document out-of-control data, the situation, and the corrective action taken.

In evaluating accuracy of the measurement process, the inspector should verify that the laboratory personnel:

- Introduce spiked samples into the train of actual samples at least 10 percent of the time to monitor the performance of the analytical system. In the spiked samples, the amount of additive is appropriate to the detection limit and sample concentration.
- Prepare and use accuracy control charts for each analytical procedure. Develop accuracy control charts by collecting data from a minimum of 15 to 20 spiked samples (run in controlled conditions) over an extended period.
 - Establish accuracy limits (as percent recovery) based on standard deviations whose upper and lower control limits are three times the standard deviation above and below the central line.
 - Establish the upper and lower warning limits at twice the standard deviation above and below the central line. Note: Some parameters have a defined warning limit required by 40 CFR Part 136.
- Take corrective actions when data fall outside the warning and control limits.
- Document out-of-control data, the situation, and the corrective action taken.

EXAMPLE OF LABORATORY QA/QC MEASURES FOR MICROBIAL ANALYSES

As an example of the laboratory quality measures an inspector might evaluate, the following discussion applies to microbial analysis. Microbial contamination is a common concern related to animal feeding operations and sanitary treatment systems covered by the NPDES standards. Common microbial contaminants of concern in wastewater and sewage sludge include total coliform, fecal coliform, and enterococci. Appropriate microbial laboratory control measures the inspector should verify include the use by laboratory personnel of:

- Positive and negative controls—controls are known cultures that are analyzed exactly like a field sample and will produce an expected positive or negative result for a given type of medium.
- Media sterility checks—media are incubated at the appropriate temperature without the field sample and observed for growth to verify the media is not contaminated with the evaluated microorganisms prior to use in the laboratory.
- Dilution sterility checks—dilution water is analyzed exactly like a field sample and observed for growth to verify the water is not contaminated with the evaluated microorganisms prior to use in the laboratory.
- Sample bottle blanks—a blank is analyzed for each bottle lot used during the sampling episode to verify the sample bottles had not been contaminated with the evaluated microorganisms prior to the field sampling.
- Membrane filter preparation blanks—membrane filter blanks are analyzed at the beginning of each set of filtered samples to verify the membrane filtration equipment is not contaminated with the evaluated microorganisms prior to use in the laboratory.
- Incubator temperature monitoring—incubator temperatures are monitored in the laboratory to verify that prepared microbial samples are being incubated at the correct temperatures.

The analytical methods for microbial analyses are specified in 40 CFR Part 136, Table IA.

EVALUATION OF PERMITTEE DATA HANDLING AND REPORTING

An analytical laboratory must have a system for uniformly recording, correcting, processing, and reporting data. The inspector should verify that the laboratory personnel:

- Use correct formulas to calculate the final results.
- Apply round-off rules uniformly.
- Establish significant figures for each analysis.
- Provide data in the form/units required for reporting.
- Ensure cross-checking calculations provisions are available.
- Determine control chart approaches and statistical calculations for the purposes of QA/QC and reporting.

- Maintain laboratory report forms that provide complete data documentation and facilitate data processing.
- Keep permanently bound laboratory notebooks or pre-printed data forms to document the procedures performed and the details of the analysis, such as the original value recorded, correction factors applied, blanks used, data values reported, personnel that performed the tests, and any abnormalities that occurred during the testing procedure.
- Define procedures for correction of data entry errors. Original data entries can be read and the individual(s) making the corrections are clearly identified.
- Back up computer data with duplicate copies (i.e., electronic and hardcopy).
- Maintain data records that allow the recalculation of all results reported by the laboratory(ies) from the original unprocessed results (i.e., raw data) to the final results sent to EPA and the regulatory authority for a minimum of three years.

EVALUATION OF PERMITTEE LABORATORY PERSONNEL

Analytical operations in the laboratory vary in complexity. Consequently, the laboratory should clearly define work assignments. All analysts should be thoroughly instructed in basic laboratory operations. Those persons performing complex analytical tasks should be qualified and properly trained. All analysts must follow specified laboratory procedures and be skilled in using the laboratory equipment and techniques required for the analyses assigned to them. In evaluating laboratory personnel, the inspector should consider the following factors:

- Adequacy of training.
- Skill and diligence in following procedures.
- Skill and knowledge in using equipment and analytical methods (particularly for complex equipment such as gas chromatography).
- Precision and accuracy in performing analytical tasks.
- Assignment of clearly defined tasks and responsibilities.

EVALUATION OF CONTRACT LABORATORIES

When the permittee contracts with the laboratory to analyze samples, the inspector may need to evaluate the laboratory practices at the contracted laboratory. The practices can also be evaluated by other designated EPA inspectors. If a deficiency is identified at a contract laboratory, the permittee is responsible for the deficiency and will be notified.

OVERVIEW OF THE DISCHARGE MONITORING REPORT QUALITY ASSURANCE PROGRAM AND HOW IT RELATES TO THE INSPECTION PROGRAM

The validity of the NPDES program depends on the quality of the self-monitoring program. The Discharge Monitoring Report Quality Assurance (DMR QA) program is an important tool used to ensure the quality of NPDES self-monitoring data. The program is designed to evaluate and improve the ability of laboratories serving NPDES permittees to analyze and report accurate self-monitoring data.

Major and selected minor permittees under the NPDES program are required to participate in the annual DMR-QA study program. DMR-QA evaluates the analytical ability of the laboratories that routinely perform self-monitoring analyses required by their NPDES permit. EPA also approves certain state laboratory certification programs to be used as either a full or a partial substitute for DMR-QA. Under the program, permittees must purchase NPDES performance evaluation samples containing constituents normally found in industrial and municipal wastewaters from accredited providers. The permittee analyzes these samples using the analytical methods and laboratory normally employed for their reporting of NPDES self-monitoring data. The supplier of the performance evaluation sample will evaluate the results and respond to the permittee.

Highlights

- The DMR-QA Program has been an excellent means of focusing on and improving the quality of laboratory results used in developing DMR data. Improvements in the DMR-QA data have been significant.
- This program has helped major permittees identify and correct both analytical and data handling problems in their laboratories.
- In general, permittees are receptive to the program and recognize its value, including some who challenged EPA's authority to require participation.
- Regions and states are generally supportive and have made good use of the results of this program for targeting inspections and directing other follow-up activities. This ability to concentrate corrective actions on problem permittees results in an increased efficiency in improving the self-monitoring data of all NPDES permittees.
- The program is one of the least resource-intensive methods for maintaining direct and regular technical contact with NPDES permittees. It has been recognized as a cost-effective effort.
- Utilizing computer technology, the following ways of managing and analyzing DMR QA data were started in fiscal year 1985: compiling tracking summaries, comparing performance of the major industries, tracking multiple permittees, and regenerating past performance evaluation reports.

The results of the DMR-QA are provided to and tracked by EPA and the state DMR-QA coordinator. The DMR-QA Program and the NPDES inspection programs are interdependent in several areas. First, EPA can use DMR-QA evaluations of permittee performance to target the inspections since the evaluations identify potential problems in the laboratory analysis or data handling and reporting. This targeting helps to direct limited resources to permittees who need them most. Non-reporting of DMR-QA results is also an important trigger for on-site inspections. Secondly, EPA can identify instances when the QA results do not comply with the parameters specified in the permit to check during the inspection.

E. REFERENCES

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

ASTM. (1982). Annual Book of Standards: Part 31, Water. Philadelphia, PA: ASTM.

- 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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F. LABORATORY QUALITY ASSURANCE CHECKLIST

| A. GENERAL | | | | |
|------------|--|--------|------------|---|
| Yes | Yes No N/A 1. Laboratory maintains a written QA/QC manual. | | | |
| B. SA | MPLE | HAND | LING | PROCEDURES |
| Yes | No | N/A | 1. | Access to laboratory area restricted to authorized personnel only. |
| Yes | No | N/A | 2. | Sample security area available within laboratory that is dry, clean, and isolated; has sufficient refrigerated space; and can be locked securely. |
| Yes | No | N/A | 3. | Laboratory refrigerator utilizes a thermometer with NIST certification or that is annually calibrated against another NIST-certified thermometer and documented using certification tags. |
| Yes | No | N/A | 4. | Laboratory has a sample custodian and a back-up custodian. |
| Yes | No | N/A | 5. | Custodian receives and logs in all incoming samples. |
| Yes | No | N/A | 6. | Custodian properly stores samples. |
| Yes | No | N/A | 7. | Custodian performs checks of proper preservation, container type, and holding times performed and documents the results. |
| Yes | No | N/A | 8. | Custodian distributes and retrieves samples to and from the analysts. |
| Yes | No | N/A | 9. | Custodian maintains chain-of-custody documentation. |
| Yes | No | N/A | 10. | Custodian and analysts ensure the minimum possible number of people handles the samples. |
| Yes | No | N/A | 11. | Custodian disposes of the samples and records upon direction of the laboratory director. |
| C. LAI | BORAT | FORY F | PROC | EDURES |
| Yes | No | N/A | 1. | EPA-approved written analytical testing procedures used and protocols are easily accessible by laboratory personnel. |
| Yes | No | N/A | 2. | If alternate analytical procedures used, proper written approval obtained. |
| Yes | No | N/A | 3. | Calibration and maintenance of instruments and equipment satisfactory. |
| Yes | No | N/A | 4. | QA procedures used. |
| Yes | No | N/A | 5. | QC procedures adequate. |
| | | | 6. | Duplicate samples are analyzed% of time. |
| | | | 7. | Spiked samples are used% of time. |
| Yes | No | N/A | 8. | Whole Effluent Toxicity (WET) testing is required by the permit and conducted by the laboratory. Culturing procedures are adequately documented for each organism tested. |
| Yes | No | N/A | 9. | WET testing protocols are clearly described. |
| Yes | No | N/A | 10. Nan | Commercial laboratory used. ne: |
| | | | Add | ress: |
| | | | Con | tact: |
| | | | Pho | ne: |

| | | | Cer | tification #: |
|--|----|-----|-----|---|
| D. LABORATORY FACILITIES AND EQUIPMENT | | | | |
| Yes | No | N/A | 1. | Adequate supply of laboratory pure water available for specific analysis. |
| Yes | No | N/A | 2. | Adequate bench, instrumentation, storage, and recordkeeping space available. |
| Yes | No | N/A | 3. | Clean and orderly work area available to help avoid contamination. |
| Yes | No | N/A | 4. | Adequate circulation and egress. |
| Yes | No | N/A | 5. | Adequate humidity and temperature control. |
| Yes | No | N/A | 6. | Adequate lighting and ventilation. |
| Yes | No | N/A | 7. | Dry, uncontaminated compressed air available. |
| Yes | No | N/A | 8. | Efficient fume hood systems available. |
| Yes | No | N/A | 9. | Adequate electrical sources available. |
| Yes | No | N/A | 10. | Instruments/equipment available and in good condition. |
| Yes | No | N/A | 11. | Vibration-free area for accurate weighing available. |
| Yes | No | N/A | 12. | Proper safety equipment (lab coats, gloves, safety glasses, goggles, and fume hoods) used when necessary. |
| Yes | No | N/A | 13. | Proper volumetric glassware used. |
| Yes | No | N/A | 14. | Glassware properly cleaned. |
| Yes | No | N/A | 15. | Written requirements for daily operation of instruments/equipment available. |
| Yes | No | N/A | 16. | Standards and appropriate blanks available to perform daily check procedures. |
| Yes | No | N/A | 17. | Sources of standards documented and where possible traceable to a national standard (e.g., NIST). |
| Yes | No | N/A | 18. | Records of each set of analysis including order in which calibration, QA/QC, and samples were analyzed are available. |
| Yes | No | N/A | 19. | Written troubleshooting procedures for instruments/equipment are available. |
| Yes | No | N/A | 20. | Written schedules for required maintenance are available. |
| Yes | No | N/A | 21. | Check the accuracy of purchased solutions as per method requirements. |
| Yes | No | N/A | 22. | Prepare stock solutions and standards using volumetric glassware. |
| Yes | No | N/A | 23. | Prepare and standardize reagents against reliable primary standards. |
| Yes | No | N/A | 24. | Use the required reagent purity for the specific analytical method. |
| Yes | No | N/A | 25. | Frequently checked working standards to determine changes in concentration or composition. |
| Yes | No | N/A | 26. | Verify concentrations of stock solutions before being used to prepare new working standards. |
| Yes | No | N/A | 27. | Background reagents and solvents run with every series of samples. |
| Yes | No | N/A | 28. | Label standards and reagents properly, including the preparation date, concentration, the analyst's identification, storage requirements, and discard date. |
| Yes | No | N/A | 29. | Store standards, reagents, and solvents in appropriate containers and under required method conditions and manufacturer's directions. |
| Yes | No | N/A | 30. | Store standards, reagents, and solvents using clean containers. |

| Yes | No | N/A | 31. | Replace gas cylinders at 100-200 psi. |
|-------------------------|-------|-------|-------|--|
| Yes | No | N/A | 32. | Written procedures exist for cleanup, hazard response methods, and applications of correction methods for reagents and solvents. |
| Yes | No | N/A | 33. | Discard standards after recommended shelf-life has expired or when signs of discoloration, formation of precipitates, or significant changes in concentrations are observed. |
| E. LA | BORAT | ORY F | REC | ISION, ACCURACY, AND CONTROL PROCEDURES |
| Yes | No | N/A | 1. | Analyzed multiple control samples (i.e., blanks, standards, duplicates, and spikes) for each type of QA/QC check and recorded information. Every tenth sample should have been followed by a duplicate and a spike. |
| Yes | No | N/A | 2. | Plotted precision and accuracy control methods used to determine whether valid, questionable, or invalid data are being generated throughout the analysis. |
| Yes | No | N/A | 3. | Taken corrective actions when data fall outside the warning and control limits. |
| Yes | No | N/A | 4. | Recorded out-of-control data, the situation, and the corrective action taken. |
| F. DA | TA HA | NDLIN | IG AI | ND REPORTING |
| Yes | No | N/A | 1. | Used correct formulas to calculate final results. |
| Yes | No | N/A | 2. | Applied round-off rules uniformly. |
| Yes | No | N/A | 3. | Established significant figures for each analysis. |
| Yes | No | N/A | 4. | Recorded data in the proper form and units for reporting. |
| Yes | No | N/A | 5. | Ensured cross-checking calculations provisions are available. |
| Yes | No | N/A | 6. | Developed and followed control chart approaches and statistical calculations for QA/QC. |
| Yes | No | N/A | 7. | Laboratory report forms developed to provide complete data documentation and to facilitate data processing. |
| Yes | No | N/A | 8. | Laboratory notebooks or pre-printed data forms bound permanently utilized to provide good documentation. |
| Yes | No | N/A | 9. | Procedures for correction of data entry errors are defined. |
| Yes | No | N/A | 10. | Backed up computer data with duplicate copies (i.e., electronic and hardcopy). |
| Yes | No | N/A | 11. | Efficient filing system exists, enabling prompt retrieval of information and channeling of report copies. |
| Yes | No | N/A | 12. | Data records allow recalculation of all results reported by the laboratory(ies) from the original unprocessed results (raw data) to the final results sent to EPA and the regulatory authority for a minimum of three years. |
| G. LABORATORY PERSONNEL | | | | |
| Yes | No | N/A | 1. | Enough analysts present to perform the analyses necessary. |
| Yes | No | N/A | 2. | Analysts have on hand the necessary references for EPA procedures being used. |
| Yes | No | N/A | 3. | Analysts trained in procedures performed through formal or informal training or certification programs. |