NOTICE OF INTENT TO ADOPT ADMINISTRATIVE RULES

TAKE NOTICE the North Dakota Department of Environmental Quality ("Department"), will hold a public hearing to address several proposed new articles to be located in Department of Environmental Quality, N.D. Admin. Code Title 33.1, on February 6, 2020 at the Environmental Training Center, 2639 East Main Avenue, Bismarck, ND.

The following shall be the hearing times: Article 33.1-12 at 10:30 a.m. to 11:30 a.m., Article 33.1-14 at 1 p.m. to 2 p.m., and Article 33.1-23 at 2:15 to 3:15 p.m. Articles 33.1-12 and 33.1-14 proposed rules are expected to have an impact on the regulated community in excess of $50,000. Article 33.1-23 proposed rules are not expected to have an impact on the regulated community in excess of $50,000. Some of the rules and other documents that are the subject of this public notice will be submitted to the United States Environmental Protection Agency (U.S. EPA).

The purpose of the proposed articles is to implement S.L. 2019, ch. 24 (H.B. 1024), moving the Boiler Inspection program to the Department from the Insurance Department; to implement S.L. 2019, ch. 24 (H.B. 1024), moving the Petroleum Tank Release Compensation Fund to the Department from the Insurance Department; and to implement S.L. 2019, ch. 217 (S.B. 2107) establishing and administering the Department's certification program for environmental laboratories.

In this rulemaking, the Department is proposing to adopt:

- Article 33.1-12, relating to petroleum tank release compensation fund
- Article 33.1-14, relating to boiler rules
- Article 33.1- 23, relating to environmental laboratory certification program rules

The proposed rules may be reviewed at the Department, 918 East Divide Ave. Bismarck, ND 58501-1947 or on the Department's website at deq.nd.gov/PublicNotice.aspx. A copy of the proposed rules may be obtained by writing to the above address or by calling 701.328.5150. The proposed rules and additional related information are also available on the Department of Environmental Quality website at deq.nd.gov/PublicNotice.aspx. Written or oral comments on the proposed rules sent to the above address, email or telephone number and received by February 18, 2020 will be fully considered.

If you plan to attend the public hearing and will need special facilities or assistance relating to a disability, please contact the Department at the above telephone number or address at least two days prior to the public hearing.

Dated this 25th day of November, 2019.

L. David Glatt, P.E.
Director
North Dakota Department of Environmental Quality

NORTH DAKOTA DEPARTMENT OF ENVIRONMENTAL QUALITY

The following chapters are created:

ARTICLE 33.1-23

DIVISION OF CHEMISTRY LABORATORIES

Chapter
33.1-23-01 Fees for Chemistry Laboratories Analysis
33.1-23-02 Environmental Laboratory Certification Program
33.1-23-03 Environmental Laboratory Certification Proficiency Testing
33.1-23-04 Environmental Laboratory Certification Fee Determination

CHAPTER 33.1-23-02

ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

Section
33.1-23-02-01 Applicability and Scope
33.1-23-02-02 Definitions
33.1-23-02-03 General Requirements for Required Methods
33.1-23-02-04 Biosolids Program Methods
33.1-23-02-05 Clean Water Act Program Methods
33.1-23-02-06 Coal Combustion Residuals Rule Program Methods
33.1-23-02-07 Non-Potable Water Program Methods
33.1-23-02-08 Potable Water Program Methods
33.1-23-02-09 Resource Conservation and Recovery Act Program Methods
33.1-23-02-10 Safe Drinking Water Act Program Methods
33.1-23-02-11 Alternate methods
33.1-23-02-12 Solids and Chemicals Program Methods
33.1-23-02-13 Personnel and Contact Information
33.1-23-02-14 Quality system
33.1-23-02-15 Access to premises
33.1-23-02-16 Access to records
33.1-23-02-17 Subcontracting
33.1-23-02-18 Certification Status
33.1-23-02-19 Response
33.1-23-02-20 Application contents for primary certification
33.1-23-02-21 Application period
33.1-23-02-22 Certification renewal
33.1-23-02-23 Modified application
33.1-23-02-24 Conditions for reapplication
33.1-23-02-25 Term of primary certification
33.1-23-02-26 Limit of certification
33.1-23-02-27 Term of reciprocal certification and application contents
33.1-23-02-28 Department notification – voluntary withdrawal or discontinuation of certification
33.1-23-02-29 Client notification required
33.1-23-02-30 Recertification

33.1-23-02-31 Certification modification, suspension, revocation, or denial

33.1-23-02-01. Applicability and Scope

This chapter applies to all laboratories required to be certified under North Dakota Century Code section 23.1-01-14. A laboratory that performs tests and analyses, the results of which must be reported to the Department to meet permit conditions or other Department program or regulatory requirements, must be certified for the parameters and methods required by the permit or Department program, unless the permit or Department program specifically exempts the parameters or methods from certification requirements. Certification requirements are equal to those required by federal programs for regulated parameters by promulgated methods unless otherwise specified or required by a Department program.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-02. Definitions.

In this article, unless the context otherwise requires, the following definitions apply:

1. “Analyte” means the chemical substance, physical property, or organism determined in a sample.

2. “Analyte Group” means a set of analytes that can be determined using the same method or technology.

3. “Biosolids” means sewage sludge or a solid, semi-solid, or liquid residue generated during the treatment of domestic sewage in a treatment works. Biosolids includes, but is not limited to, domestic septage; scum or solids removed in primary, secondary, or advanced wastewater treatment processes; and a material derived from sewage sludge. Sewage sludge does not

include ash generated during the firing of sewage sludge in a sewage sludge incinerator or grit and screenings generated during preliminary treatment of domestic sewage in a treatment works.

4. "Certified Laboratory" means a laboratory that has a valid certification issued by the Department.

5. "Client" means an entity that has arranged with a laboratory to perform tests and analyses to meet the requirements of a Department issued permit or another Department program or regulatory requirement.

6. "Coal Combustion Residual Rule" means the sampling and analysis requirements under title 40 Code of Federal Regulations, part 257 and Appendices III and IV to part 257.

7. "Department" means the North Dakota Department of Environmental Quality

8. "Department program" means a program or rule administered by the Department that requires submission of data for compliance reporting purposes that must come from a certified laboratory.

9. "Field of testing" means the combination of analyte, method, matrix, and program for which a laboratory may hold accreditation or certification

10. "Initial Application" means an application submitted by a laboratory that either has never had certification or has not met the requirements and qualifications for either a renewal or revised application.

11. "Laboratory" means a facility that performs analyses on potable water, non-potable water, a hazardous liquid or solid matrix.

12. "Manual for the Certification of Laboratories Analyzing Drinking Water" means the EPA publication Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition and including Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water and Supplement 2 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water .

13. "Method" means an EPA promulgated or EPA accepted published scientific technique for performing a specific measurement. Method includes instructions for sample preparation, sample preservation, and sample analysis.

14. "Method Defined Parameter" means parameters that are physical or chemical properties of materials determined with specific methods used to evaluate whether the materials comply with certain Resource Conservation and Recovery Act of 1976 (RCRA), 42 U.S.C. section 6901 et seq., subtitle C regulations.

15. "National Primary Drinking Water Regulations" or "NPDWR" means the federal program authorized under title 40 Code of Federal Regulations, part 141, section 141.1 et seq.

16. "National pollutant discharge elimination system" or "NPDES" means the federal program authorized under title 40 Code of Federal Regulations, part 136, section 136.1 et seq.

17. “Non-Potable Water” means water not suitable for drinking. It is a matrix in the Clean Water Act Program, the Resource Conservation and Recovery Act Program and the Coal Combustion Residuals Rule Program.

18. “North Dakota Environmental Laboratory Certification Program Manual” means the manual used by the environmental laboratory certification program for chemistry parameters. It is available on the Department’s website.

19. "Parameter" means the chemical substance, physical property, or organism being determined.

20. “Point value” means the numerical increments which represent the amount necessary to cover costs of reviewing applications, issuing certifications, conducting laboratory evaluations, training, collecting fees, and providing compliance assistance and other anticipated costs of administering the environmental laboratory certification program.

21. “Potable water” means water suitable for drinking. It is the matrix in the Safe Drinking Water Act Program.

22. "Proficiency test" means the process of testing and reporting of test results performed by a laboratory for a specific analyte or analyte group to determine the ability of a laboratory to employ applicable analytical methods and to produce an accurate measurement of the concentration of the analyte or analyte group in the sample.

23. “Reciprocal certification” means a reciprocal or secondary certification that is based on a primary certification.

24. "Renewal application" means an application submitted by a laboratory to renew an existing certification.

25. "Reporting limit" means the lowest level of an analyte that can be accurately recovered from the matrix of interest. This limit is equivalent to a level of quantitation.

26. “Resource Conservation and Recovery Act (RCRA)” means the federal law found under 42 U.S.C. section 6901 et seq. (1976) and its corresponding regulations found under title 40, Code of Federal Regulations, parts 239 through 282.

27. "Revised application" means an application that is submitted to make changes to an existing certification.

28. “SW-846” means the EPA guidance for using the “Test Methods for Evaluation Solid Waste: Physical/Chemical Methods," Publication SW-846, United States Environmental Protection Department (2019). This guidance consists of three main parts: chapters, methods and supporting documents and is the EPA SW-846 Compendium.

29. "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods " means the EPA publication also known as SW-846.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-03. General Requirements for Required Methods.

The analytical methods, sample collection, and preservation procedures used to analyze samples for programs required by a federal agency shall meet the requirements specified in the relevant parts of the Code of Federal Regulations. The laboratory's analytical methods, sample collection, and preservation procedures shall also meet the requirements specified by the Department program. Certification requirements are based on the analysis of regulated parameters by promulgated methods unless otherwise specified or required by a Department program.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-04. Biosolids Program Methods.

For analysis of sewage sludge samples required by state and federal rules, laboratories shall use the methods and test procedures in title 40, Code of Federal Regulations, part 503, and Publication SW-846.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-05. Clean Water Act Program Methods.

For analysis of water or wastewater samples required by state and federal clean water rules, laboratories shall use the methods and test procedures in title 40, Code of Federal Regulations, part 136.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-06. Coal Combustion Residuals Rule Program Methods.

For analysis of water or wastewater samples required by state and federal Coal Combustion Residuals in Landfills and Surface Impoundments rules and regulations as amended, laboratories shall use methods appropriate for groundwater sampling and that accurately measure hazardous constituents and other monitoring parameters in groundwater samples. Metals analysis must be for “total recoverable” concentrations. Parameters are found at Appendix III to part 257 and Appendix IV to part 257.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14

Law Implemented: NDCC 23.1-01-14

33.1-23-02-07. Non-Potable Water Program Methods.

For analysis of water or wastewater samples as requested by the Department or to support studies of specific industries or for use in broad national surveys, laboratories shall use validated methods and test procedures. EPA methods are preferred but other state approved and validated methods may be acceptable.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-08. Potable Water Program Methods.

For analysis of suitable drinking water samples as requested by the Department or to support studies of specific industries or for use in broad national surveys, laboratories shall use validated methods and test procedures. EPA methods are preferred but other state approved and validated methods may be acceptable.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-09. Resource Conservation and Recovery Act Program Methods.

For analysis of wastewater, waste, and solid and hazardous waste samples, laboratories shall use the methods and test procedures found in the SW-846 or as deemed by the Division of Waste Management within the Department. Modifications may be used with the approval of the Department or accrediting body except for Method Defined Parameters. Method-defined parameters can only be determined by the methods prescribed in RCRA regulations because the methods are part of the regulations. These methods must be followed exactly as written, or the resulting data cannot be used to ensure regulatory compliance. A list of method-defined parameters found under title 40 Code of Federal Regulations, part 260, section 260.11.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-10. Safe Drinking Water Act Program Methods.

For analysis of drinking water samples required by state and federal Safe Drinking Water Act rules, laboratories shall use the methods and test procedures in title 40 Code of Federal

Regulations, part 141. Laboratories also shall comply with the Manual for the Certification of Laboratories Analyzing Drinking Water requirements.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-11. Alternate methods.

Provisions for the use of alternate methods to be used in the Safe Drinking Water Act Program and the Clean Water Act program are found within the corresponding federal laws and regulations. A laboratory may request approval for alternate methods by following the instructions provided in the appropriate sections of the federal laws and regulations for the Clean Water Act Program and the Safe Drinking Water Act Program.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-12. Solids and Chemicals Program Methods.

For analysis of solids and chemical samples as requested by the Department or to support studies of specific industries or for use in broad national surveys, laboratories shall use validated methods and test procedures. EPA methods are preferred but other state approved and validated methods may be acceptable.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-13. Personnel and contact information.

A laboratory must have adequate staff with the education, training, or experience to meet the requirements of certification. At least one staff person must be identified as the laboratory administrator and that person's contact information must be provided with the certification application. The laboratory administrator must notify the Department when there are changes in contact information for the laboratory administrator, change of address, owner, or legally responsible party no later than 30 days after the change occurs.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-14. Quality system.

The laboratory must have a quality assurance and quality control program that meets the criteria specified in the North Dakota Environmental Laboratory Certification Program Manual that includes:

1. a quality assurance manual or plan;
2. standard operating procedures; and
3. traceability, documentation, record keeping, and reporting.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-15. Access to premises.

The laboratory shall allow the Department and its agents reasonable access to the laboratory for inspection and evaluation purposes and shall produce such information and records as the Department requests to determine compliance with this article.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-16. Access to records.

The laboratory shall maintain all records used to demonstrate the laboratory's compliance with certification requirements. If a laboratory analyzes samples from a client, then upon request, the laboratory shall provide to the client the records that support the client's test results. The laboratory shall also make records available to the Department upon its request.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-17. Subcontracting.

A laboratory that has samples analyzed by another laboratory shall use laboratories that have valid Department certification if the data is to be reported to the Department.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-18. Certification Status.

1. A laboratory may not alter or misrepresent its certification status and attending documents issued by the Department in any brochures, promotional literature or advertising materials. A laboratory must not describe its certification status in a manner that implies certification in areas that are outside the actual scope of certification. General statements, such as “A North Dakota Certified Laboratory” or “Certified in North Dakota” are not specific enough and can be misleading. The Department may require appropriate corrective action, including publication of a retraction of the misleading information.

2. A laboratory may not represent analytical results as certified after its certification has expired or been discontinued, suspended, or revoked.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-19. Response.

A laboratory shall timely respond in writing to any written communication from the Department.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-20. Application contents for primary certification.

1. A laboratory shall submit an initial application if:

   a. it has never received primary or reciprocal certification under this article;

   b. it has had its primary certification or Department reciprocal certification revoked in total;

   c. its certification has expired for more than one year; or

   d. it has submitted an application that has remained incomplete for more than one year.

2. To apply for initial or renewal of certification, a laboratory shall submit an application on forms provided by the Department. The required information includes:

   a. identifying information;

   b. at least one field of testing for which the laboratory seeks certification.;

   c. the laboratory’s most recent quality assurance manual or plan meeting the standards of the North Dakota Environmental Laboratory Certification Program Manual;

   d. the laboratory’s most recent standard operating procedures for each field of testing that meets the standards of the Environmental Laboratory Certification Program Manual;

   e. if the application is an initial request for certification, the most recent proficiency test result for each field of testing for which the laboratory is requesting certification. The proficiency test must have been completed no more than 12 months prior to the date that the renewal application is received by the Department, no more than 6 months prior to the date the initial application is received and must meet the proficiency test requirements.

f. a list of the laboratory's detection limits and reporting limits for each field of testing for which the laboratory is requesting certification; and

g. any other additional information requested by the Department as necessary to determine compliance in this article.

3. The owner of laboratory facilities with multiple locations shall submit a separate application for each laboratory location.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-21. Application period.

Initial applications and revised applications may be submitted to the Department at any time.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-22. Certification renewal.

When a laboratory’s certification has expired, the laboratory must apply for a renewed certification.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-23. Modified application.

A laboratory with a valid certification shall submit a modified application, including the information required for primary certifications or reciprocal certifications, to the Department to:

1. add a program for which the laboratory does not currently have certification; or

2. add a test method in a program for which the laboratory is already certified; or

3. add a parameter or analyte to a test method for which the laboratory is already certified; or

4. to change the name of the certified laboratory on the certification documents.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-24. Conditions for reapplication.

A laboratory notified of or involved in a corrective action or with a suspended certification is not eligible to apply for a certification renewal for the affected field of testing until the laboratory receives confirmation from the Department that the corrective action is complete, or the laboratory has been reinstated after suspension. If the Department revoked the laboratory's certification, the laboratory shall apply for initial certification, in accordance with this article, as if it were a new laboratory.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-25. Term of primary certification.

1. Primary certification is granted following the procedure as outlined in the North Dakota Environmental Laboratory Certification Program Manual.

2. Certification becomes effective the date of issuance and is valid for three years unless suspended, revoked, or voluntarily discontinued. Any request for an extension must be in writing. The certification period may be extended at the discretion of the Department.

3. Standards of quality in the Manual for Laboratories Analyzing Drinking Water must be met in order to qualify for certification.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-26. Limit of certification.

Certification of a laboratory is not an endorsement by the Department of the quality or validity of the data generated by a laboratory. Certification does not guarantee the usability of data generated by a laboratory for an intended purpose. The users of laboratory results are responsible for determining whether to accept or reject analytical data from a certified laboratory.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-27. Term of reciprocal certification and application contents.

1. Reciprocal certification is granted following the procedure as outlined in the North Dakota Environmental Laboratory Certification Program Manual.

2. A laboratory holding a primary accreditation or certification from another accrediting body may apply for reciprocal certification in North Dakota.

3. The Department may approve other certifying authorities of federal agencies and agencies of other states for reciprocal recognition of laboratory certification programs or portions of programs that are substantially equivalent.

4. A certification program is considered substantially equivalent if a review of the certification authority’s rules are substantially equivalent to the rules and guidelines of the Department’s laboratory certification program and including:

a. inspections of certified laboratories are performed at intervals not exceeding three years;

b. the certifying authority requires an acceptable corrective action response associated with enforcement action, suspension, or revocation from the laboratory;

c. the certifying authority is the primary authority for necessary enforcement actions, such as suspension or revocation of the laboratory's certification.

5. the Department may give reciprocal certification for a laboratory that:

a. submits an application meeting the certification requirements of section 33.1-23-02-23;

b. submits the appropriate fees with its application;

c. provides a copy of current certification documents including certificate, letter and list of certified parameters, from the primary certifying state or private or federal authority; and

d. provides a copy of the primary certifying authority's most recent audit report and including any corrective action that was taken

6. A laboratory certified under this section shall notify the Department within 30 days after any enforcement action is taken by the reciprocal certifying authority.

7. Laboratories certified under reciprocity agreements are subject to this article unless specifically stated as a unique requirement for primary certification

8. Certification becomes effective the date of issuance and is valid for up to 365 days unless suspended, revoked, or voluntarily discontinued. The certification period may be extended at the discretion of the Department. The certification period on the North Dakota certificate shall not exceed the certification period on the primary certificate.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-28. Department Notification - Voluntary Withdrawal or Discontinuation of Certification.

1. If a laboratory chooses to withdraw its application for certification or discontinue its current certification, in total or in part, the laboratory shall notify the Department in writing and specify the effective date of withdrawal or discontinuation and the field of testing for which certification is being withdrawn or discontinued. The laboratory shall submit notification at least 30 days before the effective date of withdrawal or discontinuation.

2. After the effective date of voluntary withdrawal or discontinuation of certification, the laboratory may not provide analytical results for compliance reporting or any Department program for the field of testing for which certification has been withdrawn or discontinued.

3. A laboratory shall apply for revised reciprocal certification within 30 days of the issuance of a primary revised certification.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-29. Client notification required.

1. When the laboratory is voluntarily discontinuing certification: at least 30 days before the effective date of the laboratory's discontinuation of certification, the laboratory shall notify clients and affected regulatory agencies in writing of the discontinuation date and which fields of testing will be affected. The laboratory shall submit a copy of each client notification to the Department at the same time the notification is sent under section 33.1-23-02--40.

2. The laboratory shall notify clients immediately if:

a. the primary certification authority downgrades the status for a parameter or field of testing to “not certified”; or

b. certification is revoked, suspended or terminated by the primary certification authority

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-30. Recertification.

To be recertified after voluntary withdrawal or discontinuation of certification, a laboratory shall submit an application meeting the requirements for:

1. a revised application, if reapplying within one year of the date that certification was discontinued; or

2. an initial application, if certification has been discontinued for more than one year.

History: Effective ___________, 2019.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-02-31 Certification modification, suspension, revocation, or denial.

The department may modify, suspend, revoke, or deny a certification for reasons pertaining to: circumstances which do not meet the purpose and provisions of this article, the provisions of the certification, or the materials submitted as part of the application for certification; or, violations of any applicable laws or rules. The department shall provide written notice to the laboratory specifying the basis for the modification, suspension, revocation, or denial. The laboratory may request a hearing in accordance with North Dakota Century Code chapter 28-32 on the issue of modification, suspension, revocation, or denial of the laboratory’s certification. The laboratory’s hearing request must be made in writing and received by the department within 30 days after the laboratory’s receipt of the notice.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

CHAPTER 33.1-23-03

ENVIRONMENTAL LABORATORY CERTIFICATION PROFICIENCY TESTING

Section

33.1-23-03-01. Proficiency Testing Requirements.

1. A laboratory shall successfully complete at least one proficiency test for each field of testing for which it applies for certification. The laboratory must complete the proficiency test no more than 6 months prior to submitting the application. If no proficiency test sample is available for an analyte, the laboratory is exempt from the requirements of this section only for that analyte.

2. Proficiency tests results must be included with the initial or revised certification application required for a primary certification or for reciprocal certification. Proficiency test results may also be reported to the Department by the proficiency test provider upon completion of the study.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-03-02. Laboratory testing of proficiency test study samples.

To ensure valid proficiency test results, the laboratory shall:

1. obtain all proficiency test study samples as unknowns from a nationally recognized accreditation program approved vendor;

2. manage, analyze, report, and otherwise handle all proficiency test samples in the same manner as routine samples, including the same staff, procedures, equipment, and facilities used for routine analysis for the field of testing,

3. employ the same calibration, quality control, acceptance criteria, sequence of analytical steps, number of replicates, and other standard operating procedures for proficiency test samples as used when analyzing routine samples; and

4. follow sample preparation steps for the proficiency test sample as instructed by the proficiency test sample provider.

History: Effective __________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-03-03. Reporting results.

1. A laboratory shall submit the results of all proficiency tests to the Department no later than 30 days after the laboratory receives the results from the proficiency test sample provider.

2. A laboratory conducting proficiency testing as part of an initial or revised application shall submit the results of proficiency tests as part of the application.

3. A laboratory shall either provide a copy of the original results to the Department or authorize the proficiency test sample provider to provide all results directly to the Department.

4. Proficiency test samples analyzed or reported to the proficiency test sample provider after the provider's study closing date are not valid for compliance with the proficiency testing requirements under this section.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-03-04. Restrictions on exchanging information.

Prior to the time the results of the proficiency test are submitted to the proficiency test sample provider, a laboratory may not:

1. communicate proficiency test results to another laboratory, including intercompany communication; or

2. attempt to obtain the assigned value of any proficiency test sample from a proficiency test sample provider or another laboratory.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-03-05. Evaluation of results.

1. A laboratory must demonstrate passing performance to the Department, as determined by the proficiency test sample provider, for each parameter and method or field of testing reported.

2. A laboratory may use one proficiency test sample for multiple methods.

3. A laboratory may not request a revised report from the proficiency test sample provider when the requested revisions are the result of error on the part of the laboratory.

History: Effective ___________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-03-06. Repeat proficiency tests.

1. A laboratory with primary certification may repeat proficiency tests after obtaining unacceptable results as follows:

a. if the first proficiency test result is unacceptable, the laboratory must resolve the suspected cause, document the corrective action and complete a second proficiency test within 30 days of receiving the unacceptable result;

b. if the second proficiency test result is unacceptable, the laboratory must:

(1) resolve the suspected cause and submit a corrective action report to the Department within 30 days of receiving the second unacceptable result; and

(2) order and complete a third proficiency test within 30 days of receiving the unacceptable result of the second proficiency test;

c. if the third proficiency test result is unacceptable, the laboratory may not provide analytical results for compliance reporting or any Department program for the field of testing for which the laboratory failed to demonstrate acceptable proficiency test results. The laboratory may resume providing analytical results when the laboratory passes two consecutive proficiency tests conducted at least 15 days apart. The laboratory must submit a corrective action report to the Department within 30 days of passing the second of the two proficiency tests.

2. The North Dakota Environmental Laboratory Certification Program Manual governs when a portion of a multiple analyte group proficiency test is unacceptable.

3. The Department may request additional information necessary to validate sample results generated during the testing period covered under this section.

4. A laboratory with reciprocal certification must follow the repeat proficiency test guidelines established by its primary certification Agency.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

CHAPTER 33.1-23-04

ENVIRONMENTAL LABORATORY CERTIFICATION FEE DETERMINATION

33.1-23-04-01. Method

1. Certification fees for primary initial, renewed or revised application are based on matrix, type and complexity of analytical methods that a laboratory is certified to perform.

2. Certification fees for reciprocal initial, renewed or revised applications are based on the number of fields of testing on a certificate.

3. Each fee item is assigned a point value. The cost per point value determined is multiplied by the total number of points for each application. Total applicable fees are computed by the following equation:

$X = (Y)(Z)$

Where:

X = total fees applied to the certification application invoice

Y = cost assigned to the point value in section 33.1-23-04-02

Z = sum of point values as determined for initial, renewed, or revised applications.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-04-02. Computation of cost per point value.

1. The Department computes the cost per point value based upon the salaries, wages, benefits and operating expenses.

2. The point value can be found on the Department’s website.

3. The cost associated with the point value may be adjusted on an annual basis to account for any increase in the consumer price index published by the department of labor, as of the close of the twelve-month period ending on august thirty-first of each calendar year.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14

Law Implemented: NDCC 23.1-01-14

33.1-23-04-03. Payment of fees.

1. A laboratory must pay the fees required in this article within 30 days of receiving the Department's invoice.

2. Certification of a laboratory shall not be awarded until all fees are paid.

3. Fees are nonrefundable once an invoice has been issued.

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-04-04. Initial or renewed application points.

1. The points assessed for certification application or category types designated in this subsection are multiplied by the cost per point value determined to calculate the appropriate fee

2. Application or Category Type for Primary Certifications	Point Value
a. Initial application	6
b. Renewed application	3
c. Matrix fee, aqueous	5
d. Matrix fee, solid	5
e. Matrix fee, drinking water	5
f. Electrometric assays (ion-selective electrodes)	1
g. Gravimetric Assays, Residues (solids)	1
h. Gravimetric Assays, Oil and Grease (HEM)	2
i. Titrimetric or Potentiometric Titration Assays	1
j. Colorimetric or Nephelometric Spectrophotometry	2
k. Combustion or Oxidation	3
l. IC (Ion Chromatography)	3
m. Flow Injection - Gas Diffusion – Amperometry	5
n. Waste Characteristic Extractions	2
o. Waste Characterization Assays	2
p. FLAA (Flame Atomic Absorption Spectrometry)	2
q. CVAA (Cold Vapor Atomic Absorption)	3
r. Hydride AA (Gaseous Hydride Spectrometry)	3
s. CVAFS (Cold Vapor Atomic Fluorescence Spectrometry)	3
t. TDAA (Thermal Decomposition Atomic Absorption Spectrometry)	3
u. GFAA (Graphite Furnace Atomic Absorption Spectrometry)	3
v. Ultra-Low Level Metals Assays	3
w. ICP (Inductively Coupled Plasma Emission Spectrometry)	4
x. ICP/MS (Inductively Coupled Plasma-Mass Spectrometry)	5
y. GC (Gas Chromatography)	3

z. GC/MS (Gas Chromatography-Mass Spectrometry)	4
aa. HPLC (High Performance Liquid Chromatography)	3
bb. LC/MS (Liquid Chromatography-Mass Spectrometry)	4
cc. HR-GC/MS (High Resolution GC - Mass Spectrometry)	10
dd. LC/MS/MS (Liquid Chromatography-Mass Spectrometry-Mass Spectrometry)	10
ee. WET (Whole Effluent Toxicity Assays)	5
ff. other	contact Department

3. Application or Category Type for Reciprocal Certification	Point Value
a. Initial and renewed application	10
b. Level 1: 0-200 parameters	2
c. Level 2: 201-500 parameters	5
d. Level 3: 500-1000 parameters	10
e. Level 4: 1000-2000 parameters	20

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

33.1-23-04-05. Revised applications.

1. Fees for laboratories with primary certification and applying for a revised application to add a new test category to the laboratory's certification are computed by the following equation:

X = Y + Z

Where:
X= Total fees
Y = any incurred costs for an onsite audit to review new instrumentation or technology
Z = fees associated with the point value of the new test category as outlined in the initial or renewed application points section.

2. Laboratories with primary certification and applying for a revised application to add or drop parameters under a method whereby the laboratory is already certified will be charged 1-point value to update the certificate and list of certified parameters.

3. Laboratories with reciprocal certification and applying for a revised application must pay the following:

a. fees equal to 1-point value when using the same primary certificate number as used for the initial or renewed application

b. when using a primary certificate number other than the one used for the initial or renewed application, fees are computed according to the following equation:

X = Y + Z

Where:
X = Total fees
Y = fees associated with 1-point value
Z = fees associated with the level as described in the initial or renewed application points section

History: Effective ____________, 2020.
General Authority: NDCC 23.1-01-14
Law Implemented: NDCC 23.1-01-14

Division of Chemistry
North Dakota Environmental Laboratory
Certification Program (NDELCP)
701-328-6172

North Dakota
Environmental Laboratory
Certification Program
Manual for
Chemistry Parameters

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DISCLAIMER

This manual has been prepared to describe the North Dakota Environmental Laboratory Certification Program (NDELCP). It is based on the USEPA *Manual for the Certification of Laboratories Analyzing Drinking Water - Criteria and Procedures Quality Assurance* - Fifth Edition and Supplement 1. This manual is not necessarily all inclusive of the North Dakota Environmental Laboratory Certification program and its requirements.

This document does not change or substitute for any legal requirements. While the North Dakota Environmental Laboratory Certification Program has made every effort to ensure the accuracy of the manual's discussion, the obligations of the regulated community are determined by the relevant statutes, regulations or other legally binding requirements. This manual can only reflect the regulations in place at the time of its preparation. Consequently, for any definitive description of current legal obligations, the user should not rely only on the discussion in the manual. In the event of a conflict between the discussions in this manual and any statute or regulation, this document is not controlling. The word should in this manual indicates the USEPA's and the North Dakota Department of Environmental Quality's strongly preferred approach to ensure the quality of laboratory results and may connote a requirement. The North Dakota Department of Environmental Quality may decide to revise this manual without public notice to reflect changes to its approach or to clarify and update the text.

The mention of commercial products in this manual does not constitute an endorsement of the use of that product by the USEPA or the North Dakota Department of Environmental Quality.

PREFACE

Since 1978, the U.S. Environmental Protection Agency (EPA) has implemented a certification program for laboratories performing drinking water analyses for compliance with regulations issued pursuant to the Safe Drinking Water Act. EPA recommends that States follow the criteria and procedures that EPA uses in evaluating laboratories in their own certification programs. The North Dakota Department of Environmental Quality Environmental Laboratory Certification Program uses the USEPA *Manual for the Certification of Laboratories Analyzing Drinking Water - Criteria and Procedures Quality Assurance* - Fifth Edition and Supplement 1 as the basis for its environmental laboratory certification program.

The Fifth Edition of the EPA Certification Manual was prepared to address promulgated drinking water regulations and methods; and agency policy that, at the discretion of each state's Certification Authority (CA) allows National Environmental Laboratory Accreditation Program (NELAP) accreditation to be accepted in lieu of drinking water certification in terms of producing data for compliance monitoring purposes.

Holders of this manual should check with the North Dakota Environmental Laboratory Certification Officer(s) to make sure their manual is current. Since there are no USEPA certification programs for laboratories performing analyses for compliance with regulations issued pursuant to the Clean Water Act and the Resource Conservation and Recovery Act, the North Dakota Environmental Laboratory Certification Program (NDELCP) uses the criteria in this manual as the basis for certification of laboratories performing analyses for compliance with the Clean Water Act, the Resource Conservation and Recovery Act and additional designated programs.

INTRODUCTION

North Dakota is a primacy state which means that it has primary enforcement responsibility for its drinking water regulations. The regulations at 40CFR 142.10(b)(4) require a state that has primacy to have laboratory facilities available which are certified by the EPA regional administrator. The regulations governing drinking water certification (40 CFR 141.28) require that all testing for compliance purposes be performed by certified laboratories except that drinking water testing for turbidity, free chlorine residual, temperature, pH, alkalinity, calcium, conductivity, orthophosphate, TOC, SUVA, daily chlorite, and silica may be performed by anyone acceptable to the State. Primacy states must have a program for the certification of laboratories conducting analytical measurements of drinking water contaminants pursuant to the requirements of the State primary drinking water regulations unless all analytical measurements required by the State's primary drinking water regulations are conducted at laboratories operated by the State and certified by the USEPA (40 CFR 142.10(b)(3)(i)).

North Dakota Century Code (23.1-01-14) authorizes the State Department of Environmental Quality to provide for the certification of environmental laboratories. It also stipulates, in part, that, "Unless exempted by the department , all environmental laboratories that conduct tests or prepare data for submittal to the department must be certified by the department and comply with an condition imposed by the certification."

The North Dakota Department of Environmental Quality Environmental Laboratory Certification Program for Chemistry Parameters certifies laboratories for the analysis of drinking water, wastewater, solid and hazardous waste, biosolids, potable and non-potable water to ensure compliance with requirements of the Safe Drinking Water Act, Clean Water Act, Resource Conservation and Recovery Act and additional programs respectively. Certification is not done for other purposes. Certification may be granted upon assurance that a laboratory desiring to become certified or wanting to maintain its certified status has satisfied the requirements for certification outlined in this document. The certification shall be limited to only those analyses and examinations the laboratory is capable of performing in accordance with the requirements outlined in this document.

This document describes the North Dakota Environmental Laboratory Certification Program which is based heavily on the USEPA Drinking Water Certification Program. This document is <u>not</u> a quality assurance plan and; therefore, is not to become part of any laboratory's written quality assurance plan.

FEES FOR PRIMARY CERTIFICATION

All in state laboratories are assessed fees for onsite evaluations based on incurred cost. Certification fees for Fields of Testing (FOTs) and NDELCP administrative requirements are determined by North Dakota Administrative Code 33.1-23-04. Contact the state laboratory certification officer for chemistry parameters for the current fees. Onsite evaluations are required at least once every three years in order to maintain certification.

Situations which might require an onsite evaluation more than once every three years include:

- if the laboratory requests certification to analyze additional or newly regulated contaminants, and
- if the laboratory reapplies for certification after correction of deficiencies which resulted in the downgrading or revocation of certification status.

Laboratories requiring onsite evaluations more than once every three years will be assessed fees for each visit.

Certified laboratories or laboratories seeking certification are required to purchase and analyze appropriate proficiency test samples from an American Association for Laboratory Accreditation (A2LA) or TNI accredited provider or providers.

FEES FOR RECIPROCAL CERTIFICATION

Certification fees for Fields of Testing (FOTs) and NDELCP administrative requirements are determined by North Dakota Administrative Code 33.1-23-04. Contact the state laboratory certification officer for chemistry parameters for the current fees.

LABORATORY FACILITIES

The analysis of compliance samples is to be conducted in a laboratory where the security and integrity of the samples and the data can be maintained. The laboratory facilities should be clean, have adequate temperature and humidity control, have adequate lighting at the bench top and should meet applicable OSHA standards. Meeting OSHA standards is not a requirement for certification and OSHA inspections are not part of the certification onsite evaluation process. The laboratory must have provisions for the proper storage and disposal of chemical wastes and secondary containment for hazardous waste storage is recommended. The appropriate type of exhaust hood is required where applicable.

There should be sufficient bench space for processing samples. Workbench space should be convenient to sink, water, gas, vacuum and electrical sources free of surges. Instruments should be properly grounded. For safety reasons, inorganic and organic facilities should be in separate rooms. Organic analysis and sample extraction should also be separated to prevent cross contamination. The analytical and sample storage areas should be isolated from all potential sources of contamination. There should be sufficient space for the safe storage of chemicals, glassware and portable equipment, sufficient floor space and bench space for stationary equipment and areas for cleaning materials.

LABORATORY PERSONNEL

The laboratory should have sufficient supervisory and other personnel with the necessary education, training, technical knowledge, and experience for their assigned functions.

Laboratory Director/Manager or Technical Director:

- The laboratory director/manager should be a qualified professional with the technical education and experience, and managerial capability commensurate with the size/type of the laboratory. The laboratory director/manager is ultimately responsible for ensuring that all laboratory personnel have demonstrated proficiency for their assigned functions and that all data reported by the laboratory meet the required quality assurance (QA) criteria and regulatory requirements.

Quality Assurance Manager:

- The QA manager should be independent from the laboratory management, if possible, and have direct access to the highest level of management. The QA manager should have a bachelor’s degree in science, training in quality assurance principles commensurate with the size and sophistication of the laboratory, and at least one year of experience in quality assurance. The QA manager should have at least a working knowledge of the statistics involved in quality control of laboratory analysis and a basic understanding of the methods which the laboratory employs.

Laboratory Supervisor:

- The laboratory supervisor should have at least a bachelor’s degree with a major in chemistry or equivalent, and at least one year of experience in the analysis of environmental samples. The laboratory supervisor should have at least a working knowledge of quality assurance principles. The laboratory supervisor has the responsibility to ensure that all laboratory personnel have demonstrated their ability to satisfactorily perform the analyses to which they are assigned, and that all data reported by the laboratory meet the required quality assurance and regulatory criteria.

Laboratory Analyst:

- The laboratory analyst should have at least a bachelor’s degree with a major in chemistry or equivalent, and at least one year of experience in the analysis of environmental samples. If the analyst is responsible for the operation of analytical instrumentation, he or she should have completed specialized training offered by the manufacturer or another qualified training facility or served a period of apprenticeship under an experienced analyst. The duration of this apprenticeship should be proportional to the sophistication of the instrument. Data produced by analysts and instrument operators while in the process of obtaining the required training or experience are acceptable only when reviewed and validated by a fully qualified analyst or the laboratory supervisor.

Technician:

- The laboratory technician should have at least a high school diploma or equivalent, complete a method training program under an experienced analyst and have six months of bench experience in the analysis of environmental samples. Before beginning the analysis of compliance samples, the technician must adhere to any required QC procedures specified in the method for blanks, precision, accuracy, sensitivity, specificity and satisfactory analysis on unknown samples.

Sampling Personnel:

- Personnel who collect samples should be trained in the proper collection technique for all types of samples which they collect. Their technique should be reviewed by experienced sampling or laboratory personnel.

Waiver of Academic Training Requirements:

- The certification officer may waive the need for specified academic training on a case-by case basis, for highly experienced analysts.

Training Records:

- Training records should be maintained for all personnel. These should include all job-related formal education and training taken by the analyst which pertains to any aspect of his/her responsibilities, including but not limited to analytical methodology, laboratory safety, sampling, quality assurance, data analysis, etc

ANALYTICAL METHODS

EPA approved methods must be used for analysis of each regulated Safe Drinking Water Act contaminant and each regulated Clean Water Act contaminant. There are recommended methods for analysis of Secondary drinking water contaminants. These approved and recommended methods are listed at 40 CFR Parts 141 and 142 for the Safe Drinking Water Act and at 40 CFR Part 136 for the Clean Water Act. There are EPA approved methods for analysis of disinfectant residuals and disinfection byproducts in drinking water which can be found in the Wednesday, December 16, 1998 *Federal Register (National Primary Drinking Water Regulations: Disinfectants and Disinfection Byproducts; Final Rule)* and the Wednesday, January 4, 2006 *Federal Register (National Primary Drinking Water Regulations: Stage 2 Disinfectants and Disinfection Byproducts Rule; Final Rule).* Methods for analysis of parameters for compliance with the Resource Conservation and Recovery Act are found at 40 CFR Part 261 and in *SW846 On-Line* on the EPA Web Site at www.epa.gov.

Measurements for turbidity, pH, temperature, disinfectant residual, calcium, orthophosphate, silica, alkalinity, and conductivity in drinking water need not be made by certified laboratories but may be performed by any persons acceptable to the state. However, approved methodology must be used.

- Sealed liquid secondary turbidity standards purchased from the instrument manufacturer or other sources should be calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymer standards and revised values assigned at least every four months in order to monitor for any deterioration. This calibration should be documented. These standards should be replaced when they do not fall within 15% of the initial assigned concentration of the standard. Solid turbidity standards composed of plastic, glass, or other materials are not reliable and should not be used.

- If visual comparison devices such as color wheels or sealed ampules are used for determining free chlorine residual, the standards incorporated into such devices should be calibrated at least every six months. These calibrations need to be documented. Directions for preparing temporary and permanent type visual standards can be found in Method 4500-Cl^- B, of the currently promulgated editions of *Standard Methods for the Examination of Water and Wastewater.* By comparing standards and plotting such a comparison on graph paper, a correction factor can be derived and applied to future results obtained on the now calibrated apparatus.

LABORATORY EQUIPMENT AND INSTRUMENTATION

The laboratory must have the equipment and instruments needed to perform the approved methods for which certification has been requested. All instruments are to be properly maintained and calibrated.

Balances and Weights:

- Balance range should be appropriate for the application for which it is to be used. Environmental chemistry laboratories should use balances that weigh to at least 0.0001 g. The balances should be calibrated at least annually with ASTM Type 1, Class 1 or 2 weights. (ASTM, 1916 Race St., Philadelphia, PA 19103). This may be done by laboratory personnel or under contact by a manufacturer's representative. It is strongly recommended that laboratories have a contract to calibrate balances due to the expense of the calibration weights, and to serve as an outside QC check of the weights and balances. Weights meeting ASTM Type I, Class 1 or 2 specifications should be recertified at least every five years or if there is reason to believe damage (corrosion, nicks) has occurred.

- Each day the mechanical or digital balance is used, a verification should be performed. The verification consists of a check of a reference mass at approximately the same nominal mass to be determined. Verifications should be done each weighing session. Weights meeting ASTM Type 1 specifications may be used. These should be calibrated annually against the reference weights at the time of balance calibration. The checks and their frequency should be as prescribed in the laboratory's QA plan. A record of all checks should be kept and be available for inspection.

Color Standards:

- Wavelength settings on spectrophotometers should be verified at least annually with color standards. The specific checks and their frequency should be as prescribed in the laboratory's QA documents. A record of these checks should be kept as prescribed in the laboratory's QA documents and be available for inspection.

Temperature Measuring Devices:

- Liquid bearing thermometers such as mercury or alcohol thermometers need to be traceable to NIST calibration and verified at least annually and whenever the thermometer has been exposed to temperature extremes. The correction factor should be indicated on the thermometer and the date the thermometer was calibrated and the calibration factor should be kept as prescribed in the laboratory's QA documents and be available for inspection. The NIST thermometer should be recalibrated at least every five years or whenever the thermometer has been exposed to temperature extremes.

- Digital thermometers, thermocouples and other similar electronic temperature measuring devices should be calibrated at least quarterly. The date the thermometer was calibrated, and the calibration factor should be kept as prescribed in the laboratory's QA documents and be available for inspection.

- When an infrared detection device is used to measure the temperature of samples, the device should be verified at least every six months using a NIST certified thermometer over the full temperature range that the IR thermometer will be used. This would include ambient (20 - 30^{0}C), iced (4^{0}C) and frozen (0 to -5^{0}C). Each day of use a single check of the IR thermometer should be made by checking the temperature of a bottle of water at the temperature of interest that contains a calibrated thermometer. Agreement between the two should be within 0.5^{0}C, or the device should be recalibrated.

Magnetic Stirrer:
- Variable speed with TFE coated stir bar.

pH Meter:
- Line or battery powered with accuracy of ± 0.1 units and usable with ion selective electrodes.

Conductivity Meter:
- Readable in ohms or mhos with a range of 2 ohms to 2 mhos. Line or battery operated.

Hot plate:
- Temperature controllable.

Centrifuge:
- To 3000 rpm with option of 4 x 50 mL.

Refrigerator/Freezer:
- Standard laboratory explosion proof for organics and capable of maintaining nominal temperature of 4^{0}C.

Drying Oven:
- Gravity or convection and controlled from room temperature to 180^{0}C or higher at ± 2^0 C.

Muffle Furnace:
- To 450^{0}C for cleaning organic glassware.

Glassware:
- Borosilicate and volumetric should be Class A.

Spectrophotometer:
- Range 400 - 700 nm, band width < 20 nm, use several size and shape cells with

path length 1 - 5 cm.

Filter Photometer:

- Range 400 - 700 nm, band width 10 - 70 nm, use several size and shape cells with path length 1 - 5 cm.

Amperometric Titrator

Specific Ion Meter:

- Accuracy ± 1 mV.

Inductively Coupled Plasma (sequential, simultaneous):

- Computer controlled with background correction.

Inductively Coupled Plasma/Mass Spectrometer:

- Computer controlled with background correction.
- Mass spectrometer: Range 5 - 250 amu, resolution 1 amu peak width at 5% peak height.

Water Bath:

- Electric or steam heat and controllable within 5^0C to 100^0C.

Ion Chromatograph:

- Conductivity detector, UV detector, suppression system and separator column.

Atomic Absorption Spectrophotometer:

- Single channel, single or double beam, grating monochromator, photomultiplier detector, adjustable slits, range 190 - 800 amu.
- Readout system: Response time compatible with AA, able to detect positive interference for furnace, chart recorder, CRT, or hard copy printer.
- Graphite furnace: Reach required temperatures, background corrector provision for offline analysis. Pipets and tips: microliter capacity with disposable tips, 5 - 100 microliters metal free tips.
- Arsine Generator
- Hydride Generator

Mercury Analyzer:

- Spectrophotometer: Dedicated analyzer having a mercury lamp acceptable.
- Adsorption cell: 10 cm quartz cell with quartz end windows or 11.5 cm plexiglass cell with 2.5 cm ID.
- Air pump to deliver flow of at least 1 L/min.
- Aeration tube with coarse glass frit.
- Flowmeter to measure air flow of 1L/min.
- Drying unit: 6 in tube with 20 grams magnesium perchlorate or heating device or lamp to prevent condensation on cell.

Gas Chromatograph:

- Split/splitless injection. Oven temperature control $\pm$ 0.2^{0}C. Oven temperature programmer. Sub-ambient accessory. Variable constant differential flow control.
- Electron Capture detector, linearized.
- Electrolytic Conductivity/Photoionization detector.
- Nitrogen Phosphorus detector.

Mass Spectrometer:

- Quadrupole or ion trap. All glass enrichment device, all glass transfer line, electron ionization at $\geq$ 70 eV, scanning 35 - 260 amu $\leq$ 2 sec, interfaced data system.

Purge and Trap System:

- All glass purger, 5/25 mL sample size.

High Performance Liquid Chromatograph:

- Isocratic and gradient capabilities, constant flow, capable of injecting 20 - 500 uL.
- Post column reactors.
- Detectors: Ultraviolet, fluorescence, photodiode array.

Auto Analysis System:

- Multi-channel pump, manifold, colorimeter.

GENERAL LABORATORY PRACTICES

Chemicals/reagents:

- Chemicals and reagents used must meet any requirements specified in the methods. If not specified, then "Analytical reagent grade" (AR) or American Chemical Society (ACS) grade chemicals or better should be used for analyses in certified laboratories. Consult the currently promulgated editions of *Standard Methods for the Examination of Water and Wastewater*, part 1070 for more detailed information on reagent grades. Chemicals should be dated upon receipt and upon opening to track shelf life. Reagents prepared in the laboratory need to be labeled as to what they are along with the concentration, when they were prepared, who prepared them and an expiration date.

Reagent water:

- The laboratory must have a source of reagent water having a resistance value of at least 0.5 megohms (conductivity less than 2.0 micromhos/cm) at 25^{0}C when required by the method. High quality water meeting such specifications may be purchased from commercial suppliers. Quality of reagent water is best maintained by sealing it from the atmosphere. Quality checks to ensure that the above specifications are met should be made and documented at planned intervals based on use. Individual analytical methods may specify additional requirements for the reagent water to be used. Inorganic methods require distilled or deionized water free of the analyte of interest and trace metals methods require ASTM Type 1 water. Reagent water for organic analysis must adhere to any required QC specified in the methods. Most methods specify that the reagent water does not contain analytes of interest above their respective method detection limits (MDLs). It may be necessary to treat water with activated carbon to eliminate all interferences.

Glassware preparation:

- Glassware cleaning requirements specified in the methods must be followed. If no specifications are listed, then glassware should be washed in a warm detergent solution and thoroughly rinsed, first with tap water then with reagent water. This cleaning procedure is sufficient for general analytical needs. It is advantageous to maintain separate sets of suitably prepared glassware for the nitrate and mercury analyses due to the potential for contamination from the laboratory environment.

Temperature checks:

- Temperatures of ovens, incubators, and refrigerators should be checked and documented daily or when used.

Microliter pipet calibration checks:

- The calibration of microliter pipets should be checked and documented annually.

QUALITY ASSURANCE PLAN REQUIREMENTS

All laboratories analyzing environmental compliance samples must adhere to any required quality control procedures specified in the methods. This is to ensure that routine analytical data are scientifically valid and defensible and are of known and acceptable precision and accuracy. To accomplish these goals, each laboratory should prepare a written description of its quality assurance activities, a quality assurance plan. It is the responsibility of the quality assurance manager to keep the quality assurance plan up to date. All laboratory personnel need to be familiar with the contents of the quality assurance plan. This plan should be submitted to the auditor(s) for review prior to the onsite evaluation.

The laboratory's quality assurance plan should be a separately prepared document. However, documentation for many of the listed quality assurance plan items may be made by reference to appropriate sections of this manual, the laboratory's standard operating procedures, or other literature (e.g., promulgated methods, *Standard Methods for the Examination of Water and Wastewater,* etc.). The quality assurance plan should be updated at least annually.

At a minimum, the following items should be addressed in each quality assurance plan:

- Laboratory organization and responsibility: include a chart or table showing the laboratory organization and lines of responsibility, including quality assurance managers.

- List the key individuals who are responsible for ensuring the production of valid measurements and the routine assessment of measurement systems for precision and accuracy (e.g., who is responsible for internal audits and reviews of the implementation of the plan and its requirements).

- Describe training to keep personnel updated on regulations and methodology, and document that laboratory personnel have demonstrated proficiency for the methods they perform.

- Process used to identify client's Data Quality Objectives

- SOPs with dates of last revision:
    - The laboratory should maintain SOPs that accurately reflect all phases of current laboratory activities.
    - Ensure that current copies of SOPs are in the laboratory and in the quality assurance manager's files
    - Ensure that SOPs are reviewed annually and revised as changes are made.
    - Ensure that SOPs have signature pages and revisions dated.

- Field sample procedures: describe the process used to identify sample collectors, sampling procedures and locations, required preservation, proper containers, correct sample container cleaning procedures, sample holding times from collection to analysis, and sample shipping and storage conditions.

- Ensure that appropriate forms are legibly filled out in indelible ink or hard copies of electronic data are available.

- Describe how sample are checked when they arrive for proper containers and shipping temperature preservation.

- Describe how samples are checked for proper analyte preservation (e.g., pH, chlorine residua etc.) before analysis.

- Laboratory sample receipt and handling procedures:

    - Bound laboratory notebooks, if used, should be filled out in ink; entries dated and signed. A secure, password protected, electronic data base is acceptable.
    - Store unprocessed and processed samples at the proper temperature, isolated from laboratory contaminants, standards and highly contaminated samples and, sometimes, each other; holding times may not be exceeded.
    - Maintain integrity of all samples (e.g., by tracking samples from receipt by laboratory through analysis to disposal).
    - Require Chain-of-Custody procedures for samples likely to be the basis for an enforcement action.
    - Specify criteria for rejection of samples which do not meet shipping, holding time and/or preservation requirements and procedures for notification of sample originators.

- Instrument calibration procedures:
    - Specify type of calibration used for each method and frequency of use.
    - Describe calibration standards' source, age, storage, labeling.
    - Perform data comparability checks.
    - Use control charts and for radiochemistry, report counting errors with their confidence levels.

- Analytical procedures (may reference SOP manual):
    - Cite complete method manual.
    - Describe quality control procedures required by the methods that need to be followed.

- Data reduction, validation, reporting and verification:
    - Describe the data reduction process, i.e., the method of conversion of raw data to final results.
    - Describe the data validation process.
    - Describe reporting procedures including the format.
    - Describe the data verification process.
    - For radiochemistry, describe reporting of counting uncertainties and confidence levels.
    - Describe the procedure for data corrections.

- Type of quality control checks and the frequency of their use. Parameters for chemistry and radiochemistry should include or reference:
    - Instrument performance check standards.
    - Frequency and acceptability of method detection limit (MDL) calculations.
    - Frequency and acceptability of demonstration of low-level capability.
    - Calibration, calibration verification, internal, and surrogate standards.
    - Laboratory reagent blank, field reagent blank and trip blank.
    - Field and laboratory matrix duplicates.
    - Quality control and proficiency testing samples.
    - Laboratory fortified blank and laboratory fortified sample matrix duplicates.
    - Initial demonstration of method capability.
    - Use of control charts.
    - Qualitative identification/confirmation of contaminants.

- Lists of schedules of internal and external system and data quality audits and inter laboratory comparisons.

- Preventive maintenance procedures and schedules:
    - Describe location of instrument manuals and schedules and documentation of routine equipment maintenance.
    - Describe availability of instrument spare parts in the laboratory.
    - List any maintenance contracts in place.

- Corrective action contingencies:
    - Describe response to obtaining unacceptable results from analysis of proficiency testing samples and from internal quality control checks.
    - Name staff positions responsible for the various corrective actions.
    - Describe how corrective actions taken are documented.

- Record keeping procedures:
    - Describe the procedures and documentation of those procedures.
    - List length of storage, media type (electronic or hard copy).
    - Describe security policy of electronic data bases.
    - All electronic data should have software support so it may be regenerated.

If any item is not relevant the quality assurance plan should state this and provide a brief explanation. A laboratory quality assurance plan should be responsive to the above items while remaining brief and easy to follow. Minimizing paperwork, while improving dependability and quality of data are the intended goals.

QUALITY CONTROL REQUIREMENTS

All quality control information should be readily available for inspection by auditors.

Proficiency Test (PT) Samples:

- In order to receive and maintain full certification for a Field of Testing, the laboratory must analyze a proficiency test sample (if available) at least annually for each analyte/method/program matrix for which the laboratory is certified. These proficiency test samples must be acquired from an American Association for Laboratory Accreditation (A2LA) or TNI accredited provider. Results must be within the acceptance limits established by the USEPA. The laboratory should document the corrective actions taken when a proficiency testing sample is analyzed unsuccessfully. A copy of this documentation should be available for review by the certification officer. If the results for any analyte are unacceptable, a make-up proficiency test sample must be successfully analyzed.

- Excluding vinyl chloride, the laboratory may be certified for all regulated drinking water VOCs if at least 80% of the regulated VOCs are analyzed successfully. The intention of this regulation is to allow some flexibility for random misses because the drinking water VOC methods include 20 regulated analytes. A laboratory will not be certified for an analyte which it fails repeatedly. This 80% rule for VOCs has recently been made more difficult to interpret since some PT providers are including THMs in the same vial as the VOCs. The 80% rule does not apply to the THMs.

- The Stage 1 Disinfection By Products (DBP) Rule, which became effective in January 2002, regulates the sum of five haloacetic acids (HAA5): monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, monobromoacetic acid and dibromoacetic acid. Laboratories are certified for HAA5, but successful analyses of the HAA PT samples are based on the results for the individual compounds. The 80% rule applies to the HAA5s, so if four of the 5 HAA5s are successfully analyzed, the laboratory may be certified for HAA5. As before, a laboratory will not be certified if the same analyte is failed repeatedly.

- The DBP rule also changed the way the trihalomethanes (THMs): chloroform, dichlorobromomethane, chlorodibromomethane, and bromoform PTs are evaluated. Laboratories are still certified for total THMs but under the DBP rule, each THM concentration must be reported, evaluated and passed individually to pass the PT sample. The DBP rule also states that if a laboratory fails one THM, it cannot be certified for TTHMs, but must analyze another PT sample and pass all four of the THMs in a PT sample to be certified to analyze compliance monitoring samples for total trihalomethanes.

Quality Control Samples:

- At least once each quarter, the laboratory should analyze a quality control sample for the analytes they are determining in that quarter. The sample should be prepared from a source other than that from which their working standards are prepared. The sample should be in the same concentration range as the analytical calibration curve. If errors exceed limits required by the methods, corrective action must be taken and documented, and a follow-up quality control sample needs to be analyzed as soon as possible to demonstrate that the problem has been corrected.

Calibration Curve:

- Calibration requirements in the methods must be followed. If there are no calibration requirements in the method, the following are guidelines to be used. At the beginning of each day that samples are to be analyzed, a calibration curve covering the sample concentration range and all target analytes should be generated according to the approved SOP. Depending on concentration ranges, the curve should be composed of three or more points. Field measurements (e.g. pH and chlorine residual) need to be made on instruments which have been properly calibrated as specified in the method or instrument manual and checked each day of use. The less precise the measurement, the greater the number of concentrations which should be included in the calibration curve.

Calibration Check:

- The calibration for some methods is so time-consuming that the above calibration is impractical to perform daily. When the determinative time is extensive such as Methods 508, 508.1, 515.1, 524.2, 525.2, etc. and the instrument is very stable, the calibration curve should be initially developed as specified above. Thereafter, each day analyses are performed, this curve should be verified by analysis of at least one standard for each of the target analytes at the expected concentration range. This verification should be done at both the beginning and end of the analysis period. All checks must be within the control limits required in the method or the system is to be recalibrated as specified above. The concentration of the check standard should vary from day to day across the range of analyte concentrations being measured.

- For some methods an initial conditioning injection is to be made to deactivate active sites that may have developed overnight. Depending on the method, the blank may be appropriate for this.

- Specific calibration requirements in the methods must be followed if different from the above.

- It is recommended that a calibration standard of one multicomponent analyte (PCBs, toxaphene, or chlordane) also be analyzed each day or work shift. By rotating the multicomponent analyte chosen, continuing calibration data can be obtained for all the multicomponent analytes over a period of one to two weeks. If a positive for a multicomponent analyte is found in a sample, a calibration check for the analyte should be performed as soon as possible.

Blanks:

- Requirements in the methods must be followed. A laboratory reagent blank should be carried through the full analytical procedure with every sample batch. In general, results from laboratory reagent blanks should not exceed the laboratory's minimum reporting limit, the lowest concentration of standard used for quantitation.

Laboratory Fortified Blanks:

- Requirements in the methods must be followed. LFBs should be analyzed at the level specified in the method. Some methods require that a laboratory fortified blank at ten times the MDL or a mid-level concentration be analyzed with each batch of samples. Precision and accuracy data should be documented for this determination. In addition, the analyst should routinely verify the minimum reporting limit for each analyte by analyzing a laboratory fortified blank at the minimum reporting level.

Laboratory Fortified Sample Matrix:

- Laboratory fortified sample matrix requirements in the methods must be met. If there are no laboratory fortified sample matrix requirements in the method, the following are guidelines to be used. The laboratory should add a known quantity of analytes to a percentage (to be described in the approved SOP) of the routine samples to determine sample matrix interference. The fortified concentration should not be less than the native concentration of the sample selected for fortification unless specified by the method. If the sample concentration is unknown or less than detectable, the analyst should choose an appropriate concentration (e.g. midpoint of the calibration range). Over time, samples from all routine sample sources should be fortified. The procedure should be described in the SOP. If any of these checks are not within the criteria specified in the method or within the control limits specified above, and the laboratory performance is in control, the result for that sample should be flagged to inform the data user that the results are suspect due to matrix effects.

Control Charts:

- Control charts for accuracy and precision, generated from laboratory fortified blanks should be maintained and used by the laboratory. Until sufficient data are available from the laboratory, usually a minimum of 20 to 30 test results on a specific analysis, the laboratory should use the control limits specified in the methods. If there are no control limits specified in the method, the limits may be statistically determined using the procedure below.

    - When sufficient data become available, the laboratory should develop LFB control charts from the mean percent recovery (X) and the standard deviation (S) of the percent recovery for the QC checks specified above (see Standard Methods for the Examination of Water and Wastewater, part 1020B, or similar quality control reference texts for further information). These data are used to establish upper and lower control limits as follows:

        upper control limit = X + 3S (upper warning limit + 2S)
        lower control limit = X - 3S (lower warning limit - 2S)

    - After each five to ten new recovery measurements, new control limits should be calculated using the most recent 20 - 30 data points. These calculated control limits should not exceed those established in the method. If any of these control limits are tighter than the method specifications, the laboratory should use the tighter criteria.

Initial Demonstration of Capability:

- Requirements in the methods must be followed. Before beginning the analysis of compliance samples, an initial demonstration of capability (IDC) must be performed for each method as required in the method. The IDC includes a demonstration of the ability to achieve a low background, the precision and accuracy required by the method, and determination of the method detection limit (MDL) (see below). An IDC should be performed for each instrument. As well, an IDC should be performed by each analyst for each method for which the analyst is responsible. In addition, it is recommended that the IDC also address the variability introduced if more than one sample preparation technician is used. Precision, accuracy and MDL should be similar for each technician. The analyst should re-determine IDCs when a change in the method, analyst or instrument is made which could affect the precision or accuracy or sensitivity. Minor changes should prompt a check to ascertain that the precision, accuracy and sensitivity have been maintained.

Quantitation of Multicomponent Organic Analytes (toxaphene, chlordane, and PCBs):

- The quantitation of multicomponent analytes requires professional judgment on the part of the analyst. This is required due to the complex nature of the chromatography involved, sample weathering, degradation, and interferences that may be present in the samples. The pattern of peaks found in the sample should be examined carefully and compared to a standard. The peaks in the sample that match the peak ratios in the standard can be used in quantitation. Peaks that have obvious interferences (such as pesticides or phthalates or peaks exhibiting poor peak shape) or appear to have been degraded or weathered should not be used for quantitation. A representative number (5-9) of peaks is suggested. Peak area should be used for quantitation and the analyst should ensure that the samples and standards have been integrated in the same manner. Quantitation can be done using the total peak area or height (comparing the 5-9 peaks used for quantitation of the sample to the standard) or by calculating each peak separately (using area) and taking the average concentration of the 5-9 peaks. Because of factors such as peak shape and baseline rise, the most accurate quantitation is obtained when the concentration of the sample closely matches that of the standard (e.g., within 20% of the standard). See EPA Method 8081, Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique, (EPA SW846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition) for a more detailed discussion of quantitation of multi component analytes.

- Note: For drinking water analysis, PCBs are qualitatively identified as Aroclors and measured for compliance purposes as decachlorobiphenyl. Chlordane is regulated as technical chlordane, a mixture of at least 11 major components and 30 minor ones.

Method Detection Limit (MDL) Calculation:

- Requirements in the methods must be followed. Most methods require initial MDL calculations for all analytes and certification officers should require the laboratories to calculate their detection limits for all regulated contaminants. If there is no procedure in the method to determine the detection limits, they should be determined in accordance with the procedure given in the most current revision of 40 CFR Part 136, Appendix B. The CFR requires an MDL of 0.0005 mg/L be attained for VOCs, and a MDL of 0.001 mg/L for lead if the lab will be processing source water composite samples. For organics and SOCs, a method detection limit of 1/5 of the MCL must be attained for compositing. VOCs should not be composited. The SOC detection limits listed at 40CFR (141.24(h)(18) are required to reduce monitoring.

- Sample preparation and analyses for the MDL calculation should be made over a period of at least three days to include day-to-day variation as an additional source of error. The analyst should determine MDLs initially, when any change is made which could affect the MDLs, or more frequently if required by the method.. Inorganic methods may require MDLs to be determined differently, and in all cases the methods must be followed. In addition, the analyst and laboratory must demonstrate low level capability on an ongoing basis through the ongoing MDL determination as described in the most current revision of 40 CFR 136, Appendix B, or repeated low level analyses (MRL).

- The calculation of MDLs by the CFR procedure may not be adequate for toxaphene and chlordane because they require pattern or peak recognition for identification. Presently, no standard procedure exists, so it is recommended that the MDL be defined as the lowest concentration for which pattern recognition is possible. Pattern recognition is used for qualitative identification of PCBs as Aroclors. Quantitation of PCBs is achieved by conversion of PCBs to decachlorobiphenyl (DCB).

Low Level Quantitation:

- The laboratory’s minimum reporting limits (MRL) should be reported to the client along with the data. The reporting limit must be below the MCL. Laboratories should run a LFB at their MRL every analysis day and should not report contaminants at levels less than the concentration level of their lowest standard. While this is scientifically sound practice, whether it is an acceptable practice will depend on State and Federal reporting requirements. It is important for users of data to understand the statistical and qualitative significance of the data. Laboratories may be required by the State to achieve a specific MDL or quantitation limit more stringent than that required by EPA.

RECORDS AND DATA REPORTING

Legal Defensibility:

- Compliance monitoring data should be made legally defensible by keeping thorough and accurate records. The quality assurance plan and other SOPs need to describe the policies and procedures used by the facility for record integrity, retention, and storage. If samples are expected to become a part of a legal action, chain of custody procedures should be used.

Maintenance of Records:

- Public water systems are required to maintain records of chemical analyses of compliance samples for 10 years and lead and copper for 12 years. The laboratory should maintain easily accessible records for five years or until the next certification data audit is complete, whichever is longer. Changes in ownership, mergers, or closures of laboratories do not eliminate these requirements. The client water system should be notified before disposing of records so they may request copies if needed. This includes all raw data, calculations, and quality control data. These data files may be either hard copy, microfiche or electronic. Electronic data should always be backed up by protected tape or disk or hard copy. If the laboratory changes its computer hardware or software, it should make provisions for transferring old data to the new system so that it remains retrievable within the time frames specified above. Data which is expected to become part of a legal action may need to be maintained for a longer period of time. Check with your legal counsel.

Sampling Records:

- Data should be entered in ink with any changes lined through such that the original entry is visible. Data may also be kept electronically. Changes need to be initialed and dated. The following information should be readily available:
    - Date, location (including name of utility and PWSID ID # for drinking water samples), site within the system, time of sampling, name, organization and phone number of the sampler, and analyses required.
    - For drinking water samples, identification of the sample as to whether it is a routine distribution system sample, check sample, raw or finished water sample, repeat or confirmation sample or other special purpose sample.
    - Date of receipt of the sample.
    - Sample volume/weight, container type, preservation and holding time and condition on receipt.
    - pH and disinfectant residual at time of sampling (if required) from plant records
    - Transportation and delivery of the sample (person/carrier, conditions)

Analytical Records:

- Data should be recorded in ink with any changes lined through such that the original entry is visible. Changes need to be initialed and dated. The following information should be readily available:
    - Laboratory and persons responsible for performing analyses.
    - Date and time of analysis.

  - Results of sample and quality control analyses.
  - Calibration and standards information.
  - Analyst and technician Initial Demonstration of Capability documentation should be kept on file as well as results of proficiency testing.

Reconstruction of Data:

- Adequate information should be available to allow the auditor to reconstruct the final results for compliance samples and PT samples.

SAMPLE COLLECTION, HANDLING AND PRESERVATION

Rejection of samples:

- The laboratory's rejection criteria should be documented in writing in the laboratory's QA Plan or in an SOP. The laboratory should reject any sample taken for compliance purposes which does not meet the criteria for sample containers and preservation, maximum holding times, sample collection and transport, sample collector and the sample report form. The laboratory must (141.23(a)(4)(i)) notify the authority requesting the analyses and ask for a resample. If resampling is not possible and the sample is analyzed, the sample data should be clearly identified in the data package as being unusable for its intended purpose. In addition, the inadmissibility of these sample data needs to be clearly communicated to all end data users.

Sample Containers and Preservation:

- The type of sample container and the required preservative for each inorganic and organic chemical contaminant are listed in the table below. The laboratory must measure and record the temperature of the sample when it arrives when temperature preservation is required by the method. The use of "blue ice" is discouraged because it generally does not maintain the temperature of the sample at 4°C ±2°C or less. If blue ice is used, it should be frozen at the time of sampling, the sample should be chilled before packing, and special notice taken at sample receipt to be certain the required temperature (4°C) has been maintained.

- Sample temperatures should be noted upon receipt. Samples that arrive at the laboratory within 24 hours of sample collection, due to the close proximity of a public water system to the laboratory, may not yet have reached the appropriate temperature by the time they arrive at the laboratory. These samples should be considered acceptable ONLY if packed on ice or with frozen gel/ice packs immediately after sample collection and hence, delivered while the samples were in the process of reaching an appropriate equilibrium temperature.

Maximum Holding Times:

- Samples must be analyzed within the maximum holding times required by the method. These are listed in the table below.

Sample Collection and Transport

- There must be strict adherence to correct sampling procedures, sample handling, complete identification of the sample, and prompt transfer of the sample to the laboratory when required by the method. When the laboratory is not responsible for sample collection and transport, it must verify that the paperwork, preservatives, containers and holding times are correct as required by the methods or reject the sample. The rejection criteria should (EPA Order 5360.1) be documented in writing.

Sample Collector:

- The sample collector should be trained in sampling procedures and have complete written sampling instructions (SOPs) for each type of sample to be collected. The samplers are to be able to demonstrate proper sampling technique.

Sample Report Form:

- The sample collection report form should contain, at a minimum, the ID, location, date and time of collection, collector's name, preservative added and shipping requirements, container and volume, sample type, analysis, and any special remarks concerning the sample. Indelible ink should be used.

Sample Compositing:

- If samples are composited, the compositing must (40 CFR 141.23,24) be done in the laboratory. Samples may only be composited if the laboratory detection limit is adequate for the number of samples being composited (up to a maximum of five – detection limit of 1/5 of the MCL). If the concentration of any inorganic chemical in the composite is ≥ to one-fifth of the MCL, then a follow-up sample must be taken within 14 days at each sampling point included in the composite. These samples must be analyzed for the contaminants which exceeded one-fifth the MCL in the composite sample. [CFR 144.23(a)(4)] Compositing of VOCs is not recommended.

Drinking Water
(*from 40 CFR 141.2,141.24, 141.86 and Drinking Water Cert. Manual*)

Parameter/Method	**[1]Preservative**	**[3]Sample Holding Time (Immediate = 15 minutes)**	**Extract Holding Time and Storage Conditions**	**Suggested Sample Size**	**[2]Type of Container**
Metals (except Hg)	HNO3 pH<2	6 months		1 L	P, G
Lead and Copper	HNO3 pH<2	Up to 14 days after sample collection		1 L	P,G
Mercury	HNO3 pH<2	28 days		100 mL	P, G
Alkalinity	Cool, 4C	14 days		100 mL	P, G
Asbestos	Cool, 4C	48 hours[4]		1 L	P, G
Chloride	none	28 days		100 mL	P, G
Residual Disinfectant	none	Immediate		200 mL	P, G
Color	Cool, 4C	48 hours		100 mL	P, G
Conductivity	Cool, 4C	28 days		100 mL	P, G
Cyanide	Cool, 4C Ascorbic acid (if chlorinated) NaOH pH>12	14 days		1 L	P, G
Fluoride	None	28 days		100 mL	P, G

Foaming Agents	Cool, 4C	48 hours			
Nitrate	Cool, 4C, Non-acidified	48 hours[5]		100 mL	P, G
Nitrite	Cool, 4C	48 hours		100 mL	P, G
Nitrate+ Nitrite[6]	H2SO4 pH<2	28 days		100 mL	P, G
Odor	Cool, 4C	24 hours		200 mL	G
pH	None	Immediate		25 mL	P, G
o-Phosphate	Cool, 4C	48 hours		100 mL	P, G
Silica	Cool, 4C	28 days		100 mL	P
Solids (TDS)	Cool, 4C	7 days		100 mL	P, G
Sulfate	Cool, 4C	28 days		100 mL	P, G
Temperature	None	Immediate		1 L	P, G
Turbidity	Cool, 4C	48 hours		100 mL	P, G
502.2	Sodium Thiosulfate or Ascorbic Acid, 4C, HCl pH<2	14 days		40-120 mL	Glass with PTFE Lined Septum
504.1	Sodium Thiosulfate Cool, 4C,	14 days	4C, 24 hours	40 mL	Glass with PTFE Lined Septum
505	Sodium Thiosulfate Cool, 4C	14 days (7 days for Heptachlor)	4C, 24 hours	40 mL	Glass with PTFE Lined Septum
507	Sodium Thiosulfate Cool, 4C, Dark	14 days(see method for exceptions)	4C, dark 14 days	1 L	Amber Glass with PTFE Lined Cap
508	Sodium Thiosulfate Cool, 4C, Dark	7 days (see method for exceptions)	4C, dark 14 days	1 L	Glass with PTFE Lined Cap
508A	Cool, 4C	14 days	30 days	1 L	Amber Glass with PTFE Lined Cap
508.1	Sodium Sulfite HCl pH<2 Cool, 4C	14 days (see method for exceptions)	30 days	1 L	Glass with PTFE Lined Cap
515.1	Sodium Thiosulfate or Sodium Sulfite HCl pH<2 Cool, 4C, Dark	14 days	4C, dark 28 days	1 L	Amber Glass with PTFE Lined Cap

515.2	Sodium Thiosulfate or Sodium Sulfite HCl pH<2 Cool, 4C, Dark	14 days	≤4C, dark 14 days	1 L	Amber Glass with PTFE Lined Cap
515.3	Sodium Thiosulfate Cool, 4C, Dark	14 days	≤4C, dark 14 days	50 mL	Amber Glass with PTFE Lined Cap
515.4	Sodium Sulfite, dark, cool ≤10C for first 48 hr; ≤6C thereafter	14 days	≤0C 21 days	40 mL	Amber glass with PTFE Lined septum
524.2	Ascorbic Acid or Sodium Thiosulfate HCl pH<2, Cool 4C	14 days		40-120 mL	Glass with PTFE Lined Septum
525.2	Sodium Sulfite, Dark, Cool, 4C, HCl pH<2	14 days (see method for exceptions)	≤4C 30 days	1 L	Amber Glass with PTFE Lined Screw Cap
525.3	L-Ascorbic Acid, EDTA trisodium salt, Potassium dihydrogen citrate, Cool to ≤6°C	14 days, ≤6° C but not frozen (see method for exceptions)	-5°C in dark, 28 days (see method for exceptions)	1 L or 1 quart	Amber Glass with PTFE lined screw cap
531.1, 6610	Sodium Thiosulfate, Monochloroacetic acid, pH<3, Cool, 4C	Cool 4C 28 days		60 mL	Glass with PTFE Lined Septum
531.2	Sodium Thiosulfate, Potassium Dihydrogen Citrate buffer to pH 4, dark, ≤10C for first 48 hr, ≤6C thereafter	28 days		40 mL	Glass with PTFE Lined Septum

536	Ammonium acetate and sodium omadine, Cool to ≤6°C	28 days		40 mL	Amber Glass with PTFE lined screw cap
547	Sodium Thiosulfate Cool, 4C	14 days(18 mo.frozen)		60 mL	Glass with PTFE Lined Septum
548.1	Sodium Thiosulfate (HCl pH 1.5-2 if high biological activity) Cool, 4C, Dark	7 days	≤4C 14 days	≥250 mL	Amber Glass with PTFE Lined Septum
549.2	Sodium Thiosulfate, (H2SO4 pH<2 if biologically active) Cool, 4C, Dark	7 days	21 days	≥250 mL	High Density Amber Plastic or Silanized Amber Glass
550,550.1	Sodium Thiosulfate Cool, 4C, HCl pH<2	7 days	4C, Dark 550, 30 days 550.1, 40 days	1 L	Amber Glass with PTFE Lined Cap
551.1	Sodium Sulfite, Ammonium Chloride, pH 4.5-5.0 with phosphate buffer Cool, 4C	14 days		≥ 40 mL	Glass with PTFE Lined Septum
552.1	Ammonium chloride Cool, 4C, Dark	28 days	≤4C, dark 48 hours	250 mL	Amber Glass w/ PTFE Lined Cap
552.2	Ammonium chloride Cool, 4C, Dark	14 days	≤4C, dark 7 days ≤-10C 14 days	50 mL	Amber Glass with PTFE Lined Cap
555	Sodium Sulfite HCl, pH≤2 Dark, Cool 4C	14 days		≥ 100 mL	Glass with PTFE Lined cap
1613	Sodium		Recommend	1 L	Amber

	Thiosulfate Cool, 0-4C, Dark		40 days		Glass with PTFE Lined Cap

[1] For cyanide determinations samples must be adjusted with sodium hydroxide to pH 12 at the time off collection. When chilling is indicated the sample must be shipped and stored at 4 °C or less. Acidification of nitrate or metals samples may be with a concentrated acid or a dilute (50% by volume) solution of the applicable concentrated acid. Acidification of samples for metals analysis is encouraged and allowed at the laboratory rather than at the time of sampling provided the shipping time and other instructions in Section 8.3 of EPA Methods 200.7 or 200.8 or 200.9 are followed.

[2] P = plastic, hard or soft; G = glass, hard or soft.

[3] In all cases samples should be analyzed as soon after collection as possible. Follow additional (if any) information on preservation, containers or holding times that is specified in method.

[4] Instructions for containers, preservation procedures and holding times as specified in Method 100.2 must be adhered to for all compliance analyses including those conducted with Method 100.1.

[5] If the sample is chlorinated, the holding time for an unacidified sample kept at 4 °C is extended to 14 days

[6] Nitrate-Nitrite refers to a measurement of total nitrate.

Clean Water Act
(from 40 CFR 136.3 Table II—Required Containers, Preservation Techniques, and Holding Times)

Parameter number/name	**Container**[1]	**Preservation**[2 3]	**Maximum holding time**[4]
Table IA—Bacterial Tests:			
1-5. Coliform, total, fecal, and *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	8 hours.[22 23]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	8 hours.[22]
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	8 hours.[22]
8. *Salmonella*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	8 hours.[22]
Table IA—Aquatic Toxicity Tests:			

9-12. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C[16]	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C[18]	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C[18]	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C[18]	48 hours.
10. Boron	P, FP, or Quartz	HNO_3 to pH <2	6 months.
11. Bromide	P, FP, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, FP G	Cool, ≤6 °C[18]	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
16. Chloride	P, FP, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes.
21. Color	P, FP, G	Cool, ≤6 °C[18]	48 hours.
23-24. Cyanide, total or available (or CATC) and free	P, FP, G	Cool, ≤6 °C[18], NaOH to pH >10[5] [6], reducing agent if oxidizer present	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH <2	6 months.
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
Table IB—Metals:[7]			
18. Chromium VI	P, FP, G	Cool, ≤6 °C[18], pH = 9.3-9.7[20]	28 days.
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH <2	28 days.

35. Mercury (CVAFS)	FP, G; and FP-lined cap[17]	5 mL/L 12N HCl or 5 mL/L BrCl[17]	90 days.[17]
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except boron, chromium VI, and mercury	P, FP, G	HNO_3 to pH <2, or at least 24 hours prior to analysis[19]	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C[18]	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C[18]	48 hours.
41. Oil and grease	G	Cool to ≤6 °C[18], HCl or H_2SO_4 to pH <2	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤6 °C[18], HCl, H_2SO_4, or H_3PO_4 to pH <2	28 days.
44. Orthophosphate	P, FP, G	Cool, to ≤6 °C[18] [24]	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
49. Phosphorous (elemental)	G	Cool, ≤6 °C[18]	48 hours.
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
53. Residue, total	P, FP, G	Cool, ≤6 °C[18]	7 days.
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C[18]	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C[18]	7 days.
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C[18]	48 hours.
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C[18]	7 days.

61. Silica	P or Quartz	Cool, ≤6 °C[18]	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C[18]	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C[18]	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C[18], add zinc acetate plus sodium hydroxide to pH >9	7 days.
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes.
68. Surfactants	P, FP, G	Cool, ≤6 °C[18]	48 hours.
69. Temperature	P, FP, G	None required	Analyze.
73. Turbidity	P, FP, G	Cool, ≤6 °C[18]	48 hours.
Table IC—Organic Tests:[8]			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5]	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9]	14 days.[9]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$, pH to 4-5[10]	14 days.[10]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols[11]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$	7 days until extraction, 40 days after extraction.
7, 38. Benzidines[11 12]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5]	7 days until extraction.[13]
14, 17, 48, 50-52. Phthalate esters[11]	G, FP-lined cap	Cool, ≤6 °C[18]	7 days until extraction, 40 days after extraction.
82-84. Nitrosamines[11 14]	G, FP-lined cap	Cool, ≤6 °C[18], store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction.

88-94. PCBs[11]	G, FP-lined cap	Cool, ≤6 °C[18]	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone[11]	G, FP-lined cap	Cool, ≤6 °C[18], store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction.
1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons[11]	G, FP-lined cap	Cool, ≤6 °C[18], store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers[11]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction.
29, 35-37, 63-65, 107. Chlorinated hydrocarbons[11]	G, FP-lined cap	Cool, ≤6 °C[18]	7 days until extraction, 40 days after extraction.
60-62, 66-72, 85, 86, 95-97, 102, 103. CDDs/CDFs[11]			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], pH <9	1 year.
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C[18]	7 days.
Tissue Samples: Field Preservation	G	Cool, ≤6 °C[18]	24 hours.
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤−10 °C	1 year.
114-118. Alkylated phenols	G	Cool, <6 °C, H_2SO_4 to pH <2	28 days until extraction, 40 days after extraction.
119. Adsorbable Organic Halides (AOX)	G	Cool, <6 °C, 0.008% $Na_2S_2O_3$ HNO_3 to pH <2	Hold *at least* 3 days, but not more than 6 months.

120. Chlorinated Phenolics		Cool, <6 °C, 0.008% $Na_2S_2O_3$ H_2SO_4 to pH <2	30 days until acetylation, 30 days after acetylation.
Table ID—Pesticides Tests:			
1-70. Pesticides[11]	G, FP-lined cap	Cool, ≤6 °C[18], pH 5-9-[15]	7 days until extraction, 40 days after extraction.
Table IE—Radiological Tests:			
1-5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH <2	6 months.
Table IH—Bacterial Tests:			
1. *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5]	8 hours.[22]
2. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	8 hours.[22]
Table IH—Protozoan Tests:			
8. *Cryptosporidium*	LDPE; field filtration	1-10 °C	96 hours.[21]
9. *Giardia*	LDPE; field filtration	1-10 °C	96 hours.[21]

[1] “P” is for polyethylene; “FP” is fluoropolymer (polytetrafluoroethylene (PTFE); Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; “G” is glass; “PA” is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); “LDPE” is low density polyethylene.

[2] Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (e.g., using a 24-hour composite sample; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately, and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the

compositing procedure produces results equivalent to results produced by arithmetic averaging of results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirement of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater; Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under Sec. 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0-6 °C, with minimum head space.

[5] ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g., chlorine). Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (20th and 21st editions) addresses dechlorination procedures.

[6] Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365-09a. There may be interferences that are not mitigated by the

analytical test methods or D7365-09a. Any technique for removal or suppression of interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365-09a or the analytical test method must be documented along with supporting data.

[7] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (*i.e.,* use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 30 days at <0 °C.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[16] Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples

arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.

[17] Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[18] Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤°C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

[19] An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

[20] To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

[21] Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

[22] Sample analysis should begin as soon as possible after receipt; sample incubation must be

started no later than 8 hours from time of collection.

[23] For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB-EC) or 1681 (A-1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

[24] The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (*i.e.,* that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (*i.e.,* within 15 minutes of collection).

PROCEDURE FOR OBTAINING PRIMARY CERTIFICATION

The laboratory certification process begins with the laboratory director making a formal written request to the laboratory certification officer for chemical parameters. The application may apply to one of the following instances:

- A request for first-time certification for chemical parameters;
- A request for certification to analyze additional or newly regulated contaminants;
- A request to reapply for certification after correction of deficiencies which resulted in the revocation of certification status for a parameter or group of parameters.

The formal written request must be sent to the Laboratory Certification Officer for Chemical Parameters, North Dakota Department of Environmental Quality, Division of Chemistry, Box 5573, Bismarck, ND 58506-5573 and should include the following:

- Name and mailing address of the laboratory.
- Physical location of the laboratory if different from the mailing address.
- Contact person at the laboratory who is responsible for managing certification issues and this individual's telephone number.
- A list of the environmental monitoring program(s): Safe Drinking Water Act, Clean Water Act, Resource Conservation and Recovery Act, Biosolids, Coal Combustion Residuals Rule, Potable and Non-Potable Water and the respective parameters for which certification is desired.
- A list of the EPA approved methods for the analysis of each program parameter for which certification is being sought.
- A summary of the results of analysts' initial demonstration of capability tests for analysis of each parameter by each method for which certification is being sought.
- A list of laboratory instrumentation including manufacturer and model numbers.
- A copy of the laboratory's quality assurance plan.
- Copies of the laboratory's written methods for the analysis of the parameters for which certification is being sought if these are not included in the quality assurance plan.
- A list of personnel including qualifications and experience.
- A general description of the facility including approximate square footage and layout.

The state laboratory certification officer for chemical parameters will respond to a formal request for certification as soon as possible and preferably within thirty days. The response will consist of an acknowledgment of the request for certification and a request for the laboratory to obtain and analyze appropriate proficiency test samples covering the parameters requested for certification in each program area for which certification is being requested. The proficiency test samples must be obtained from an American Association for Laboratory Accreditation (A2LA) or TNI accredited provider.

The laboratory will need to analyze and report results for the proficiency test samples within the deadline established by the provider. If the laboratory's results for any parameters for which certification is desired are not acceptable, the laboratory will need to take appropriate corrective action and participate in additional proficiency testing, analyzing at least those parameters for which certification is desired but for which initial proficiency testing was not-acceptable. Failure to perform acceptably on this follow-up proficiency testing will require a comprehensive corrective action program and re-application for certification including a summary of what steps were taken to correct the deficiencies before any further proficiency testing will be accepted. In such cases limited technical assistance will be provided to the laboratory if requested.

When the proficiency testing requirements are satisfactorily completed, the state laboratory certification officer for chemical parameters will send a contract for payment of the fee for the onsite evaluation for signature(s), dating and return. Once the signed and dated certification fee contract is returned to the certification officer, the certification officer will contact the laboratory to schedule an onsite evaluation.

The onsite evaluation will consist of:

- a comprehensive review of laboratory facilities;
- general good laboratory practices;
- sample collection, preservation, and handling;
- laboratory equipment and instrumentation;
- analytical capabilities (proper adherence to approved methods);
- quality control procedures;
- documentation;
- data handling and reporting;
- record keeping; etc.

Evaluation will use criteria outlined elsewhere in this document. A written report of the evaluation will be issued within 45 days if possible and will include at a minimum the following:

- The date of inspection.
- A list of personnel consulted at the laboratory.
- A summary of what was evaluated and the findings of the evaluation.
- A list of deficiencies which must be corrected and time limits for making the corrections.
- Remarks, including recommended improvements
- Signature(s) of person(s) who performed the inspection.

Certification status for each Field of Testing may or may not be included in the report. In all cases, a certificate will be issued along with an accompanying list of certified parameters and the method(s) by which each parameter is certified in each program area. Certification status is determined according to the following ratings:

- Certified: The laboratory meets the minimum regulatory performance criteria explained in this manual and the *USEPA Manual for the Certification of Laboratories Analyzing Drinking Water - Criteria and Procedures Quality Assurance, Fifth Edition* as well as all other applicable regulatory requirements.

- Provisionally Certified: The laboratory has deficiencies but has demonstrated its ability to consistently produce valid data within the acceptance limits specified in the appropriate regulations. A provisionally certified laboratory may analyze samples for compliance purposes if the laboratory's clients are notified of the provisional status, in writing, on any report.

- Not Certified: The laboratory possesses major deficiencies and, in the opinion of the Certification Officer, cannot consistently produce valid results.

- Interim Certification: This type of certification may be granted in certain circumstances when it is impossible or unnecessary to perform an on-site audit. An example of a situation to which this type of certification is applicable is when a laboratory has requested certification for the analysis of additional analytes that involve a method for which the laboratory already has certification. In such a case, the certification officer will have already determined that the laboratory has the appropriate instrumentation, is using the approved methods, and has adequately trained personnel to perform the analyses. Satisfactory analysis of the additional analyte or analytes on a proficiency test sample, if available, is required. An onsite audit including the additional analyte or analytes will be done no later than the next regularly scheduled onsite audit.

Laboratories must not misrepresent their certification status in any brochures, promotional literature or advertising materials. It is the ethical responsibility of certified laboratories to describe their certification status in a manner that does not imply certification in areas that are outside the actual scope of certification. General statements, such as "A North Dakota Certified Laboratory" or "Certified in North Dakota" are not specific enough and can be misleading. Improper references to certification status may be dealt with by suitable actions which could include legal or corrective action or publication of a retraction of the misleading information.

Any certified laboratory which sub-contracts analytical work to another laboratory must establish that the contracted laboratory has been certified by the North Dakota Environmental Laboratory Certification Program for the appropriate parameters. Laboratory records and reports must clearly indicate who performed the analyses and the name of the contract laboratory must be included in those records and reports.

REQUIREMENTS FOR MAINTAINING CERTIFICATION STATUS

In order to maintain full certification status for analysis of each Field of Testing, laboratories certified for environmental chemical contaminants must satisfactorily analyze appropriate proficiency test samples, at least annually. These proficiency test samples must be acquired from an American Association for Laboratory Accreditation (A2LA) accredited or TNI provider. Proficiency test samples should be analyzed in the same manner as routine samples and by the person(s) who routinely analyze environmental compliance samples.

In order to maintain certification for parameters under the Safe Drinking Water Act, laboratories must use the EPA approved methods for determining compliance with the Safe Drinking Water Act. In order to maintain certification for parameters under the Clean Water Act, laboratories must use the EPA approved methods for determining compliance with the Clean Water Act. In order to maintain certification for parameters under the Resource Conservation and Recovery Act, laboratories must use EPA SW846 methods. EPA approved methods for the analysis of drinking water are specified in the drinking water regulations at 40 CFR part 141. EPA approved methods for the analysis of wastewater are specified in the Clean Water Act regulations at 40 CFR part 136.

In order to maintain certification, laboratories must pass an onsite evaluation at least once every three years. However, if the laboratory undergoes a major change or repeatedly fails a proficiency test sample, an onsite evaluation may be conducted before the usual thee year period has expired.

Certified laboratories should notify the certification officer, in writing, within 30 days of major changes in personnel, equipment, or laboratory location that might impair the laboratory’s analytical capability. A major change in personnel is defined as the loss or replacement of the laboratory supervisor or a situation in which a trained and experienced analyst is no longer available to analyze a particular Field of Testing for which certification has been granted. The certification officer will discuss the situation with the laboratory supervisor and establish a schedule for the laboratory to address major changes. If the certification officer determines that the laboratory can no longer produce valid data, action will be initiated to revoke the laboratory’s certification.

CRITERIA AND PROCEDURES FOR DOWNGRADING / REVOKING CERTIFICATION STATUS

A certified laboratory will have its certification status for a contaminant or group of contaminants downgraded to “provisionally certified” for any of the following reasons:

- Failure to analyze a proficiency test sample at least annually within the acceptance limits specified in the regulations, or, if there are no requirements specified in the regulations, within policy described by the certifying authority. The laboratory is permitted only three opportunities during each calendar year to report acceptable results for each FOT on the appropriate proficiency test studies from an American Association for Laboratory Accreditation or TNI accredited provider.
- Failure to notify the certification officer, in writing, within thirty days of major changes in personnel, equipment, or laboratory location.
- Failure to satisfy the certification officer that the laboratory is maintaining the required standard of quality, based on an onsite evaluation.
- Failure to report compliance data to the public water system or the State drinking water program in a timely manner, thereby preventing compliance with Federal or State regulations and endangering public Department of Environmental Quality. Data which may cause the system to exceed an MCL should be reported as soon as possible.

If a laboratory is subject to downgrading on the basis of the above criteria, the certification officer should notify the laboratory director or owner (by registered or certified mail or email with received/read receipt) of the intent to downgrade within 14 days from becoming aware of the situation warranting downgrading. The laboratory director will have thirty days in which to review the problems cited and send a letter to the certification officer specifying what immediate corrective actions are being taken and any proposed actions that need the concurrence of the certification officer. The certification officer should consider the adequacy of the response and notify the laboratory, in writing (by registered or certified mail or email with received/read receipt) of its certification status within 14 days of receipt of the response.

If a laboratory fails to analyze a proficiency test sample or other unknown sample within the acceptance limits, the laboratory’s certification status should not be downgraded if the laboratory resolves the suspected cause, documents the corrective action and completes a second proficiency test within thirty days of receiving the unacceptable result. If the laboratory analyzes this second unknown sample within the acceptance limits established by the USEPA or State, the laboratory’s certification status should not be downgraded. If the laboratory fails to analyze this second proficiency test sample within the established limits, the laboratory must resolve the suspected cause, submit a corrective action report to the certification officer and order and complete a third proficiency test within thirty days of receiving the unacceptable result. The certification officer should downgrade the laboratory’s certification status to “provisionally certified” and notify the laboratory within 14 days (by registered or certified mail or email with received/read receipt). The laboratory's certification status should be downgraded for only the failed analyte, except where the EPA/State certifies a group of related analytes based on a limited number of analytes in a group. If the third proficiency test result is unacceptable, the laboratory may not provide analytical results for compliance reporting of any Department program for the FOT for which the laboratory failed to demonstrate acceptable proficiency test results. The laboratory may resume providing analytical results when the laboratory passes two consecutive proficiency tests conducted at least 15 days apart. The laboratory must submit a corrective action report to the Department within 30 days of passing the second of the two proficiency tests.

During any phase of this procedure, a laboratory may request that the State provide technical assistance to help identify and resolve any problem.

If the certification officer notifies a laboratory, in writing, that it has been downgraded to "provisionally certified" status for procedural, administrative, equipment or personnel deficiency, the laboratory should correct its problem within three months.

A provisionally certified laboratory may continue to analyze samples for compliance purposes but should notify its clients of its downgraded status and provide that information, in writing, on any report.

A laboratory should be downgraded from certified, provisionally certified or interim certified status to "not certified" for a FOT analysis for the following reasons:

- Reporting proficiency test data from another laboratory as its own.
- Falsification of data or other deceptive practices.
- Failure to use the analytical methodology specified in the regulations.
- For provisionally certified laboratories, failure to successfully analyze a proficiency test sample or other unknown test sample for the FOT within the acceptance limits specified.
- For provisionally certified laboratories, failure to satisfy the certification officer that the laboratory has corrected deviations identified during an onsite evaluation.
- For provisionally certified laboratories, persistent failure to report compliance data to the public water system or the State drinking water program in a timely manner thereby preventing compliance with Federal and/or State regulations and endangering public Department of Environmental Quality. Data which may cause the system to exceed an MCL should be reported as soon as possible.
- Refusal to participate in an onsite evaluation conducted by the certification officer.

If a laboratory is to have its certification status for one or more parameters revoked, the certification officer should notify the laboratory, in writing (by registered or certified mail or email with received/read receipt), of the intent to revoke certification. If the laboratory wishes to challenge this decision, a notice of appeal should be submitted in writing to the certification officer within thirty days of receipt of the notice of intent to revoke certification. The notice of appeal should be supported with an explanation of the reasons for the challenge and should be signed by a responsible official from the laboratory such as the president/owner for a commercial laboratory, or the laboratory supervisor in the case of a municipal laboratory.

If no notice of appeal is filed, certification should be revoked. If a notice of appeal is filed, the certification officer should decide within thirty days of receipt of the appeal and notify the laboratory in writing (by registered or certified mail or email with received/read receipt). Denial of the appeal should result in immediate revocation of the laboratory's certification. Once certification is revoked, a laboratory may not analyze environmental samples for compliance purposes until its certification has been reinstated.

If the appeal is determined to be valid, the certification officer should take appropriate measures to reevaluate the facility and notify the laboratory, in writing (by registered or certified mail or email with received/read receipt), of this decision within thirty days of the reevaluation.

Through a written request, a laboratory may seek upgrading or reinstatement of certification, when and if the laboratory can demonstrate to the certification officer's satisfaction that the deficiencies which produced provisionally certified status or revocation have been corrected. This may include an onsite evaluation, successful analysis of unknown samples or any other measure the certification officer deems appropriate.

PROCEDURE FOR OBTAINING RECIPROCAL CERTIFICATION

The North Dakota Environmental Laboratory Certification Program (NDELCP) provides reciprocal certification based upon a primary certification provided by an approved organization such as A2LA, NELAP (TNI) and EPA Region certification.

Programs included in the reciprocal certification are Safe Drinking Water Act, Clean Water Act, Resource Conservation and Recovery Act (RCRA) based on SW 846 compendium, Biosolids 40 Part 506 and the Coal Combustion Residuals Rule.

Certification under these programs is granted only for parameters that are regulated and analyzed by promulgated methods.

Certification for Potable Water, Non-potable water and the Solids programs is granted for parameters analyzed by EPA accepted or Department approved methods.

Reciprocal certification is granted to in-state laboratories as well as out of state laboratories. Reciprocal certification will only be granted for the dates on a valid certificate and must be renewed every 365 days.

The main requirements for maintaining the recognition of certification are (1) that the NDELCP be notified, in writing, within thirty days of any changes in the status of the Laboratory's certification for the parameters by the methods on the NDELCP list of certified parameters during the effective period of the recognition of certification; and (2) that the NDELCP be sent copies of the reports of the Laboratory's participation in proficiency test studies for the parameters by the methods on the enclosed list during the effective period of the recognition of certification.

If the laboratory desires to renew certification with North Dakota when the recognition of certification expires, an authorized representative will need to contact the NDELCP to initiate the renewal process.

The procedure for obtaining reciprocal certification is posted on the Departments website and is as follows:

- The North Dakota Department of Environmental Quality does not directly certify out-of-state laboratories. The Department will consider recognition of out-of-state laboratories certifications or accreditations by EPA regional offices or other state certification programs on a case-by-case basis. Laboratories desiring recognition of their primary certifications or accreditations from other programs need to contact the certification officer (see below) to request an application form, a contract for payment of the fee and the fee schedule. In addition to submitting a completed application, fee contract and the fee, made payable to the North Dakota Department of Environmental Quality, laboratories must submit, at a minimum, the following information:

- A copy of the report(s) of the most-recent audit of the laboratory by the certification program(s) whose certification(s) the laboratory wishes recognized by the North Dakota Environmental Laboratory Certification Program. The laboratory must have an onsite audit by the certification program(s) at least once every three years.

- A copy of the laboratory's response(s) to the audit report(s).

- A copy of the certification(s) issued by the certification program(s) whose certification(s) the laboratory wishes recognized by the North Dakota Environmental Laboratory Certification Program. This (these) certification(s) must include dates of issue and expiration and a list of the parameters and methods for which the laboratory is certified in each program area.

- A copy of the reports of the laboratory's participation in the two most-recent proficiency test studies for each program for which recognition of certification is desired, unless these have been sent previously.

- A list, prepared by the laboratory, including the parameters, method(s) and the method source for each program for which recognition of certification is desired. The laboratory must have certification for these parameters and methods from the appropriate certification authority or authorities as listed on the application. For the SDWA program, certification will be recognized for only those parameters requiring certification by the USEPA and certified by the current EPA approved methods and any additional parameters for which the North Dakota Safe Drinking Water Program requires certification, which must also be certified by the current EPA approved methods. For the CWA program, certification will be recognized for only those parameters listed in the method tables in 40 CFR Part 136 that are certified by the current EPA approved methods listed in the tables or that are certified by methods for which EPA Region 8 has issued Alternate Test Procedure Approval. For the RCRA program, certification will be recognized for only those parameters listed in and certified by the current promulgated SW846 methods. For Potable Water, Non-Potable Water and Solids programs certification is given for parameters analyzed by EPA or Department approved methods.

If the primary certifying authority's certification program has not been reviewed by the North Dakota laboratory certification officer, the laboratory will be requested to send a copy of the primary certifying authority's rules and regulations. If the primary certifying authority's certification program has been deemed to be substantially equivalent to the North Dakota certification program, and if the laboratory submits the required information and fee payment, the laboratory’s certification may be recognized by the North Dakota Environmental Laboratory Certification Program.

To initiate the certification process and obtain an application and a contract for payment of the fee, please contact the laboratory certification officer at 701-328-6172 or at the following address:

Laboratory Certification Officer
ND Department of Environmental Quality
Division of Chemistry
P.O. Box 5537
Bismarck, ND 58502-5537

LABORATORY ETHICS AND FRAUD DETECTION / DETERRENCE

Laboratories are encouraged to have an ethics policy and implement a fraud detection and deterrence policy/program, including use of the following, as appropriate:

- data validation and verification techniques; and
- analyst notation and sign-off on manual integration changes to data.

Prohibited practices include, but are not limited to:

- fabrication, falsification, or misrepresentation of data;
- improper clock setting or improper date/time recording;
- unwarranted manipulation of samples, software, or analytical conditions;
- misrepresenting or misreporting quality control samples;
- improper calibrations;
- concealing a known analytical or sample problem;
- concealing a known improper or unethical behavior or action;
- failing to report the occurrence of a prohibited practice or known improper or unethical act to the appropriate laboratory representative or contract representative, or to an appropriate government official.

Table of Secondary Drinking Water Standards

Contaminant	Secondary MCL	Noticeable Effects above the Secondary MCL
Aluminum	0.05 to 0.2 mg/L*	colored water
Chloride	250 mg/L	salty taste
Color	15 color units	visible tint
Copper	1.0 mg/L	metallic taste; blue-green staining
Corrosivity	Non-corrosive	metallic taste; corroded pipes/ fixtures staining
Fluoride	2.0 mg/L	tooth discoloration
Foaming agents	0.5 mg/L	frothy, cloudy; bitter taste; odor
Iron	0.3 mg/L	rusty color; sediment; metallic taste; reddish or orange staining
Manganese	0.05 mg/L	black to brown color; black staining; bitter metallic taste
Odor	3 TON (threshold odor number)	"rotten-egg", musty or chemical smell
pH	6.5 - 8.5	low pH: bitter metallic taste; corrosion high pH: slippery feel; soda taste; deposits
Silver	0.1 mg/L	skin discoloration; graying of the white part of the eye
Sulfate	250 mg/L	salty taste
Total Dissolved Solids (TDS)	500 mg/L	hardness; deposits; colored water; staining; salty taste
Zinc	5 mg/L	metallic taste

*mg/L is milligrams of substance per liter of water.

Proposed UCMR 4 analytical methods and contaminants

Assessment Monitoring (List 1 contaminants)

The proposed Assessment Monitoring sampling time frame addresses the period of March 2018 through November 2020. See table below for the proposed sampling design.

National Sample Assessment Monitoring Design

System Size (# of people served)	10 List 1 Cyanotoxins	20 Additional List 1 Chemicals
Small Systems (25 – 10,000)	800 randomly selected surface water (SW) or ground water under the direct influence of surface water (GWUDI) systems	800 randomly selected SW, GWUDI and ground water (GW) systems
Large Systems (10,001 and over)	All SW or GWUDI systems	All SW, GWUDI and GW systems

- Total for small systems is additive because these systems would only be selected for one component of UCMR 4 sampling (10 cyanotoxins or 20 additional chemicals). EPA would pay for all analytical costs associated with monitoring at small systems.
- The number of large systems is not additive. All SW and GWUDI systems would monitor for cyanotoxins; those same systems would also monitor for the 20 additional List 1 chemicals, as would the large GW systems.

Ten Cyanotoxin Chemical Contaminants[3,4]

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical Methods
total microcystin	N/A	0.3 µg/L	EPTDS and SR	ELISAExit
microcystin-LA	96180-79-9	0.008 µg/L	EPTDS	EPA 544
microcystin-LF	154037-70-4	0.006 µg/L	EPTDS	EPA 544
microcystin-LR	101043-37-2	0.02 µg/L	EPTDS	EPA 544
microcystin-LY	123304-10-9	0.009 µg/L	EPTDS	EPA 544
microcystin-RR	111755-37-4	0.006 µg/L	EPTDS	EPA 544
microcystin-YR	101064-48-6	0.02 µg/L	EPTDS	EPA 544
Nodularin	118399-22-7	0.005 µg/L	EPTDS	EPA 544
anatoxin-a	64285-06-9	0.03 µg/L	EPTDS	EPA 545
cylindrospermopsin	143545-90-8	0.09 µg/L	EPTDS	EPA 545

Two Metals

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical MethodsThe following links exit the site Exit
Germanium	7440-56-4	0.3 µg/L	EPTDS	EPA 200.8, ASTM D5673-10, SM 3125
Manganese	7439-96-5	0.4 µg/L	EPTDS	EPA 200.8, ASTM D5673-10, SM 3125

Eight Pesticides and One Pesticide Manufacturing Byproduct

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical Methods
alpha-hexachlorocyclohexane	319-84-6	0.01 µg/L	EPTDS	EPA 525.3
Chlorpyrifos	2921-88-2	0.03 µg/L	EPTDS	EPA 525.3
Dimethipin	55290-64-7	0.2 µg/L	EPTDS	EPA 525.3
Ethoprop	13194-48-4	0.03 µg/L	EPTDS	EPA 525.3
Oxyfluorfen	42874-03-3	0.05 µg/L	EPTDS	EPA 525.3
Profenofos	41198-08-7	0.3 µg/L	EPTDS	EPA 525.3
tebuconazole	107534-96-3	0.2 µg/L	EPTDS	EPA 525.3
total permethrin (cis- & trans-)	52645-53-1	0.04 µg/L	EPTDS	EPA 525.3
Tribufos	78-48-8	0.07 µg/L	EPTDS	EPA 525.3

Three Brominated Haloacetic Acid (HAA) Groups[4,5]

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical Methods
HAA5	N/A	N/A	Stage 2 DBPR and/or DSMRT	EPA 552.3 or EPA 557
HAA6Br	N/A	N/A	Stage 2 DBPR and/or DSMRT	EPA 552.3 or EPA 557
HAA9	N/A	N/A	Stage 2 DBPR and/or DSMRT	EPA 552.3 or EPA 557

Three Alcohols

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical Methods
1-butanol	71-36-3	2.0 µg/L	EPTDS	EPA 541
2-methoxyethanol	109-86-4	0.4 µg/L	EPTDS	EPA 541

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical Methods
2-propen-1-ol	107-18-6	0.5 µg/L	EPTDS	EPA 541

Three Other Semivolatile Chemicals

Contaminant	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical Methods
butylated hydroxyanisole	25013-16-5	0.03 µg/L	EPTDS	EPA 530
o-toluidine	95-53-4	0.007 µg/L	EPTDS	EPA 530
Quinoline	91-22-5	0.02 µg/L	EPTDS	EPA 530

Indicators

Parameter	CAS Registry Number[1]	Minimum Reporting Level	Sampling Points[2]	Analytical MethodsThe following links exit the site Exit
Total Organic Carbon (TOC)	N/A	N/A	EPTDS	SM 5310 B, SM 5310 C, SM 5310 D (21st edition), or SM 5310 B-00, SM 5310 C-00, SM 5310 D-00 (SM Online), EPA Method 415.3 (Rev. 1.1 or 1.2)
Bromide	N/A	N/A	EPTDS	EPA Methods 300.0 (Rev. 2.1), 300.1 (Rev. 1.0), 317.0 (Rev. 2.0), 326.0 (Rev. 1.0) or ASTM D 6581-12
Temperature	N/A	N/A	EPTDS	SM 2550 (21st edition), or SM 2550-10 (SM Online)
pH	N/A	N/A	EPTDS	EPA Method 150.1 and 150.2, ASTM D1293-12, SM 4500-H+ B (21st edition) or SM 4500-H+ B-00 (SM Online)

Notes

1. Chemical Abstract Service (CAS) Registry Number
2. Sampling Locations
    1. Entry points to the distribution system (EPTDS)
    2. Source Water Intake Locations (SR)
    3. Stage 2 Disinfectants and Disinfection Byproduct Rule Sampling Locations (Stage 2 DBPR)
    4. Distribution system maximum residence time (DSMRT)
3. Temperature and pH must be measured at the same time as cyanotoxin sample collection at the source water intake.
    1. pH methods include: EPA Method 150.1 and 150.2, ASTM D1293-12, SM 4500-H+ B (21st edition) or SM 4500-H+ B-00 (SM Online).
    2. Temperature methods include: SM 2550 (21st edition), or SM 2550-10 (SM Online).
4. TOC and bromide samples must be collected at the same time as HAA samples. These indicator samples must be collected at a single source water intake using methods already approved for compliance monitoring.
    1. TOC methods include: SM 5310 B, SM 5310 C, SM 5310 D (21st edition), or SM 5310 B-00, SM 5310 C-00, SM 5310 D-00 (SM Online), EPA Method 415.3 (Rev. 1.1 or 1.2).
    2. Bromide methods include: EPA Methods 300.0 (Rev. 2.1), 300.1 (Rev. 1.0), 317.0 (Rev. 2.0), 326.0 (Rev. 1.0) or ASTM D 6581-12.
5. Regulated HAAs (HAA5) are included in the proposed monitoring program to gain a better understanding of co-occurrence with currently unregulated disinfection byproducts.
    1. HAA5 includes: dibromoacetic acid, dichloroacetic acid, monobromoacetic acid, monochloroacetic acid, trichloroacetic acid.
    2. HAA6Br includes: bromochloroacetic acid, bromodichloroacetic acid, dibromoacetic acid, dibromochloroacetic acid, monobromoacetic acid, tribromoacetic acid.
    3. HAA9 includes: bromochloroacetic acid, bromodichloroacetic acid, chlorodibromoacetic acid, dibromoacetic acid, dichloroacetic acid, monobromoacetic acid, monochloroacetic acid, tribromoacetic acid, trichloroacetic acid.