

Chapter NR 149

LABORATORY ACCREDITATION

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Note: Chapter NR 149 as it existed on April 30, 2008, was repealed and a new chapter NR 149 was created, Register April 2008 No. 628, effective September 1, 2008. Chapter NR 149 as it existed on June 28, 2021, was repealed and a new chapter NR 149 was created, Register February 2021 No. 782, effective June 29, 2021.

Subchapter I — General Provisions

NR 149.01 Purpose. The purpose of this chapter is to establish a program for the accreditation of laboratories performing testing under s. 299.11, Stats.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.02 Applicability. (1) This chapter specifies requirements for the administration of the laboratory accreditation program by the department.

(2) Unless otherwise exempted in this section, this chapter applies to all the following:

- (a) Laboratories applying for accreditation.
- (b) Laboratories holding an accreditation.
- (c) Laboratories submitting data to the department for a covered program.
- (d) Laboratories generating data that is necessary for the department to determine compliance with a covered program.

Note: Administrative codes and covered programs requiring analyses to be performed by an accredited laboratory are chs. NR 101 – Reports And Fees For Wastewater Discharges, 102 – Water Quality Standards For Wisconsin Surface Waters, 106 – Procedures For Calculating Water Quality Based Effluent Limitations For Point Source Discharges To Surface Waters, 110 – Sewerage Systems, 123 – Well Compensation Program, 131 – Nonferrous Metallic Mineral Prospecting, 132 – Nonferrous Metallic Mineral Mining, 140 – Groundwater Quality, 150 – Environmental Analysis And Review Procedures, 153 – Targeted Runoff Management And Notice Of Discharge Grant Programs, 155 – Urban Nonpoint Source Water Pollution Abatement And Storm Water Management Grant Program, 157 – Management Of Pcb's And Products Containing Pcb's, 182 – Nonferrous Metallic Mineral Mining Wastes, 200 – Application For Discharge Permits And Water Quality Standards Variances, 204 – Domestic Sewage Sludge Management, 205 – General Provisions,

206 – Land Disposal Of Municipal And Domestic Wastewaters, 210 – Sewage Treatment Works, 211 – General Pretreatment Requirements, 212 – Waste Load Allocated Water Quality Related Effluent Limitations, 214 – Land Treatment Of Industrial Liquid Wastes, By-Product Solids And Sludges, 216 – Storm Water Discharge Permits, 217 – Effluent Standards And Limitations For Phosphorus, 218 – Method And Manner Of Sampling, 219 – Analytical Test Methods And Procedures, 230 – Inorganic Chemicals Manufacturing, 233 – Pesticide Chemicals, 243 – Animal Feeding Operations, 254 – Iron And Steel Manufacturing, 256 – Metal Molding And Casting, 260 – Electroplating, 261 – Metal Finishing, 263 – Coil Coating, 273 – Nonferrous Metals Forming And Metal Powders, 274 – Nonferrous Metals Manufacturing, 290 – Steam Electric Power Generating, 347 – Sediment Sampling And Analysis, Monitoring Protocol And Disposal Criteria For Dredging Projects, 500 – General Solid Waste Management Requirements, 507 – Environmental Monitoring For Landfills, 518 – Landspreading Of Solid Waste, 528 – Management Of Accumulated Sediment From Storm Water Management Structures, 538 – Beneficial Use Of Industrial Byproducts, 662 – Hazardous Waste Generator Standards, 664 – Hazardous Waste Treatment, Storage And Disposal Facility Standards, 665 – Interim License Hazardous Waste Treatment, Storage And Disposal Facility Standards, 700 – General Requirements, 712 – Personnel Qualifications For Conducting Environmental Response Actions, 716 – Site Investigations, 720 – Soil Cleanup Standards, 738 – Temporary Emergency Water Supplies, 747 – Petroleum Environmental Cleanup Fund, 809 – Safe Drinking Water, 810 – Requirements For The Operation And Maintenance Of Public Water Systems, 811 – Requirements For The Operation And Design Of Community Water Systems, 812 – Well Construction And Pump Installation and 845 – County Administration Of NR 812 Private Wells Code.

Note: Links to the codes specified above can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(3) Laboratories performing analyses for the safe drinking water program under ch. NR 809 or for the well construction and pump installation testing program under ch. NR 812 shall be certified; registration is not available for these analyses. Additional requirements for laboratories performing compliance analysis under ch. NR 809 are specified in s. NR 149.19.

(4) Laboratories performing analysis for whole effluent toxicity testing shall meet the requirements specified in s. NR 149.20.

(5) This chapter applies to laboratories analyzing industrial pre-treatment samples when the department is the control author-

ity of a pre-treatment ordinance or when another control authority requires it.

(6) Laboratories performing asbestos or radiological testing for a covered program shall be certified or approved by the EPA or the department.

Note: Laboratories performing bacteriological testing for a covered program are certified or approved under ch. ATCP 77 by the department of agriculture, trade, and consumer protection.

(7) (ag) This chapter establishes compliance requirements that shall be incorporated into the quality systems of all laboratories accredited by the department.

(ar) Laboratories shall meet any requirements pertaining to analyses and analytical operations contained in the methods, regulations, or covered programs when those requirements are more stringent than the ones specified in this chapter, unless this chapter grants explicit, alternative allowances.

Note: Sources, including the following as updated, likely contain methods that are acceptable for testing under this chapter: The EPA, the department, Standard Methods for the Examination of Water and Wastewater, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods – SW-846, American Society for Testing and Materials, and the U.S. Geological Survey Agency.

(b) When it is not apparent to the laboratory whether the minimum requirements of this chapter or those specified in the methods, regulations, or covered programs are more stringent, the department shall communicate which requirements are more stringent to the laboratories and the laboratories shall meet those requirements.

(c) When a laboratory incorporates a procedure that is neither expressly permitted nor prohibited by the method, the department will assess the scientific validity of the procedure to determine if the procedure is within the scope of the method. The underlying chemistry of the method shall remain unchanged. The department may seek the advice of the council in making determinations under this paragraph.

Note: For example, if a digestion time of 30 minutes is required, the laboratory is not to use less time for digestion. A second example, when a minimum volume of solvent is required to extract a specific amount of sample, then less solvent is not to be used unless the ratio of extraction solvent to sample amount is maintained.

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NR 149.03 Definitions. In this chapter:

(1) “Acceptance limits” means limits established by the department that are used to determine if a laboratory has analyzed a proficiency testing sample successfully.

Note: Acceptance limits are specified in s. NR 149.27.

(2) “Accreditation” and “accredited” mean that the department has determined that an organization is competent to perform specific types of tests. “Accreditation” and “accredited” include “certification” and “registration.”

(3) “Accreditation matrix” means a matrix type that is part of the first tier of a field of accreditation under s. NR 149.13 (2). Accreditation matrices are drinking water, aqueous, and non-aqueous matrices.

(4) “Accuracy” means the closeness of a measured value to an accepted reference value or standard.

(5) “Algorithm” means a process or set of rules to be followed in calculations for solving a problem.

(6) “Analysis day” means the day in which a specific type of analysis is performed.

(7) “Analyte” means the chemical substance, physical property, or organism analyzed in a sample.

(8) “Analyte group” means a set of analytes that can be determined using the same method or technology and that constitute a unit, acknowledged by the department, of the third tier of accreditation under s. NR 149.13 (4).

(9) “Analytical balance” means a balance that is capable of measuring masses to within 0.0001 g.

(10) “Analytical class” means a set of analytes or analyte groups of similar behavior or composition, or a set of analytes or analyte groups regulated under the same provisions of the federal safe drinking water act, that is used to organize the third tier of accreditation under s. NR 149.13 (4).

(11) “Analytical instrument” means any test instrument used to provide analytical results that is not support equipment.

(12) “Analytical staff” means staff that includes laboratory directors, supervisory personnel, quality assurance personnel, technicians, chemists, biologists, preparation analysts, and instrument analysts.

(13) “Aqueous” means an accreditation matrix that is water, is not drinking water, and can be reported in units of mass per volume.

Note: Leachates are not accredited under the aqueous matrix.

(14) “Batch” means a set of environmental samples prepared or analyzed together using the same process, personnel, and lots of reagents.

(a) A “preparation batch” means a set of one to 20 environmental samples of the same accreditation matrix, meeting batch criteria, and with a maximum time of 24 hours between the start of processing of the first and last sample in the batch.

(b) An “analytical batch” means a set of environmental samples which are analyzed together as a group in an uninterrupted sequence.

(15) “Bias” means the consistent deviation of measured values from a true value caused by systematic errors in a procedure or a measurement process.

(16) “Calibration” means the process used to establish an observed relationship between the response of an analytical instrument and a known amount of analyte, or the process used to determine, by measuring or comparison with a reference standard, the correct value of each scale reading in an instrument, meter, or measuring device.

(17) “Calibration blank” means an aliquot that consists of the same matrix as that used for the calibration standards, but without the analytes.

(18) “Calibration function” means the specific mathematical relationship established to relate calibration standards to instrument response.

(19) “Calibration model” means an algorithm that is used to determine an average calibration factor, average response factor, linear regression, or non-linear regression.

(20) “Certificate” means a document owned by the department and issued to a laboratory that indicates the fields of accreditation granted to a laboratory.

(21) “Certification” or “certified” means certification, under s. 299.11 (7), Stats., of laboratories that perform compliance analyses for hire or to laboratories that perform compliance drinking water analyses in accordance with the standards and requirements of this chapter.

(22) “Coefficient of determination” means a quantity that measures the degree of agreement between the points in a calibration and the function derived to connect the points.

(23) “Confirm” means to verify the identity of a compound by an alternative procedure, column, detector, wavelength, or by a technology that bases detection on a different scientific principle from the one originally used for identifying the compound.

(24) “Continuing calibration blank” or “CCB” means an aliquot that consists of the same matrix as that used for the cali-

bration standards, but without the analytes, analyzed during an analysis sequence to verify the continued absence of instrumental interferences.

(25) “Continuing calibration verification standard” or “CCV standard” means a standard of known concentration of analyte used to assure continued calibration accuracy during an analysis sequence.

(26) “Correlation coefficient” means a quantity that measures the degree of agreement between the points in a calibration curve and the linear function derived to connect the points.

(27) “Corrective action” means any measure taken to eliminate or prevent the recurrence of the causes of an existing non-conformity, defect, or undesirable condition.

(28) “Council” means the certification standards review council created under s. 15.107 (12), Stats.

(29) “Covered program” means a program listed or enumerated in s. 299.11 (1) (d) 1. to 9., Stats., and includes any department program, project, permit, contract, or site investigation that requires analytical work to be performed by an accredited laboratory.

Note: The note in s. NR 149.02 (2) (d) provides a list of department administrative rules of covered programs requiring accreditation under this chapter.

(30) “Deficiency” means a documented or verifiable deviation from the requirements of this chapter that is noted during an on-site evaluation or while reviewing analytical data produced by a laboratory.

(31) “Department” means the department of natural resources.

(32) “EPA” means the United States environmental protection agency.

(33) “Field of accreditation” means a 3-tiered unit by which the department uses to grant laboratories accreditation as specified under s. NR 149.13.

(34) “For hire” means offering analyses for payment or non-monetary compensation.

(35) “Initial calibration blank” or “ICB” means an aliquot that consists of the same matrix as that used for the calibration standards, but without the analytes, analyzed following the initial calibration and prior to quantitating any samples to verify the absence of instrumental interferences.

(36) “Initial calibration verification standard” or “ICV standard” means a standard of known concentration, prepared using second source standards, analyzed following the initial calibration and prior to quantitating any samples to assure initial calibration accuracy.

(37) “Internal standard” means a known concentration of standard added to a sample or quality control sample as a reference for evaluating and controlling the precision and bias of the analytical method.

(38) “Laboratory” means a facility that performs tests in connection with a covered program that requires data from an accredited laboratory. A facility consisting of a principal laboratory and annexes within 5 miles of the principal laboratory may be considered a single laboratory. When the terms “laboratory” or “laboratories” are used unmodified in this chapter, the terms include laboratories accredited under this chapter and those seeking accreditation under this chapter.

(39) “Laboratory control sample” or “LCS” means a sample of a matrix without the analytes of interest or a matrix with a consistent concentration of the analytes of interest, fortified with a verified known amount of the analytes of interest. The purpose of an LCS is to determine whether the methodology is in control

and whether the laboratory can make accurate and precise measurements.

Note: In many EPA methods, the term “lab-fortified blank” is equivalent to an LCS.

(40) “Laboratory equipment” means any support equipment or analytical instrument necessary to or involved in generating the results of an analysis.

(41) “Limit of detection” or “LOD” means the lowest concentration or amount of analyte that can be identified, measured, and reported with confidence that the concentration is not a false positive value. The department considers the LOD to be equivalent to the method detection limit and is determined under the method cited in sub. (46).

(42) “Limit of quantitation” or “LOQ” means the lowest concentration or amount of an analyte for which quantitative results can be obtained.

(43) “Maximum contaminant level” or “MCL” means the maximum permissible level of a contaminant in water that is delivered to any user of a public water system.

(44) “Method” means a procedure used for measuring the presence and concentration of physical and chemical pollutants.

(45) “Method blank” means a clean matrix that is treated and processed exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates to measure artifacts in the measurement process.

Note: In many EPA methods, the term “laboratory reagent blank” is equivalent to a method blank.

(46) “Method detection limit” or “MDL” means the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. The MDL is generated according to the procedure specified in the latest revision of 40 CFR Part 136, Appendix B.

Note: Links to 40 CFR Part 136, Appendix B can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(47) “NIST” means the National Institute for Standards and Technology.

(48) “Non-aqueous” includes all matrices that are not drinking water or aqueous. It includes soils, sediments, sludges, organic liquids, oils, solid waste, and multi-phasic wastes. Leachates are accredited under the non-aqueous matrix.

(49) “Nonconformance” means a documented or verifiable deviation from the requirements of this chapter or a deviation from the requirements of a quality system.

(50) “On-site evaluation” means an assessment conducted by the department at a laboratory seeking or maintaining accreditations to determine actual or potential compliance with the requirements of this chapter.

(51) “Ownership” means owning or controlling, directly or indirectly, a laboratory facility through an equity interest, or its equivalent, of 10% or more.

(52) “Pesticide” means a chemical substance defined in s. 94.67 (25) and (25m), Stats., an isomer of a pesticide or a degradation product or metabolic product of a pesticide.

(53) “Precision” means the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as the standard deviation, variance, or range, in either absolute or relative terms.

(54) “Proficiency testing sample” or “PT sample” means a sample obtained from an approved proficiency testing sample provider to evaluate the ability of a laboratory to produce an analytical test result meeting the definition of acceptable perfor-

mance outlined in s. NR 149.27. The concentration of the analyte in the sample is unknown to the laboratory at the time of analysis.

(55) “Qualify” means a written statement accompanying or referencing test results to identify anomalies or deviations from the requirements in this chapter that were encountered in generating the results.

(56) “Quality assurance” means an integrated system of activities involving planning, control, assessment, reporting, and improvement to ensure that a product or service meets defined standards of quality.

(57) “Quality control” means the overall system of technical activities designed to measure and control the quality of a product or service that meets the stated needs of users.

(58) “Quality control limit” means the acceptance criteria used to evaluate quality control samples. Quality control limits may be those published by the department, referenced in a method or calculated by a laboratory. In this chapter, quality control limits calculated by a laboratory will be referred to as generated in-house control limits.

(59) “Quality system” means a structured and documented management arrangement describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services.

(60) “Raw data” means any original information from a measurement activity or study recorded in media that allows the reconstruction and evaluation of the activity or study. “Raw data” include absorbance, emission counts, area counts, peak heights, abundance, and millivolts. Raw data may be stored in hard copy or electronically.

(61) “Reagent water” means water which has been treated to remove any impurities that may affect the quality of an analysis.

(62) “Reference material” means a material that has one or more sufficiently well-established properties that can be used for calibrating or verifying the calibration of support equipment or analytical instruments.

(63) “Registration” or “registered” means registration under s. 299.11 (8), Stats., of laboratories that perform tests solely on its own behalf or that of a subsidiary under common ownership or control in accordance with the standards and requirements of this chapter. Registered laboratories do not perform drinking water testing.

(64) “Relative standard error” or “RSE” means the standard error divided by the mean for a set of calibration data and expressed as a percentage. The RSE is calculated according to the following formula:

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{n - p}}$$

x_i = true amount of analyte in calibration level i , in mass or concentration units.

x'_i = measured amount of analyte in calibration level i , in mass or concentration units.

p = number of terms in the fitting equation.

Note: average = 1, linear = 2, quadratic = 3, cubic = 4
 n = number of calibration points.

(65) “RV” means relative value.

(66) “Relocation” means a move by a laboratory resulting in a change in the laboratory’s physical address.

(67) “Replicate” means two or more substantially equal aliquots analyzed independently for the same analyte.

(68) “Residual” means the difference, expressed as a percent, between the theoretical concentration of a calibration standard and the value derived from the calibration function from the measured response of the calibration standard.

(69) “Result” means the quantitative or qualitative output of an analysis, including measurements, determinations, and information obtained or derived from tests.

(70) “Revocation” means cancellation of a laboratory’s accreditation.

(71) “Second source standard” means a standard procured from a supplier or manufacturer different from the supplier or manufacturer of a laboratory’s calibration standards, or a standard obtained from the same supplier or manufacturer of a laboratory’s calibration standards from a lot verifiably different from the lot of the calibration standards.

(72) “Sensitivity” means the capability of a method or instrument to discriminate between measurement responses representing different levels of analyte, or the capability of a method or instrument to detect an analyte at or greater than a stated quantity.

(73) “Shall” means a mandatory requirement.

(74) “Subcontract” means the act of procuring analytical services from a certified laboratory.

Note: Registered laboratories only do testing for their own facility. Another facility, under the same ownership, can procure analytical services from a registered laboratory.

(75) “Support equipment” means devices that may not be analytical instruments, but that are necessary to support laboratory tests and operations. “Support equipment” includes autoclaves, balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, sample preparation devices, and volumetric dispensing devices when quantitative results depend on the accuracy of the support equipment.

(76) “Surrogate” means a substance unlikely to be found in environmental samples, with properties similar to those of analytes of interest, which is used to evaluate the bias of an analysis in the fortified sample.

(77) “Suspension” means a temporary cancellation of a laboratory’s certification.

(78) “Temperature blank” means a sample container, of at least 40 mL capacity, filled with water and transported with each shipment of collected samples to determine the temperature of other samples in the shipment on arrival at a laboratory.

(79) “Test” means any chemical, biological, physical, radiological, or microscopic assay, examination, or analysis conducted by a laboratory on water, wastewater, groundwater, a biosolid, a waste material, a hazardous substance, or any other matrix analyzed to determine compliance with a covered program.

(80) “X-intercept” means the point at which the plot of the calibration function crosses the x-axis.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (38), (48), (60) made under s. 35.17, Stats., Register February 2021 No. 782.

Subchapter II — Program Administration

NR 149.05 Required accreditation. (1) All laboratories submitting data to the department for a covered program or generating data to determine compliance with a covered program, shall be accredited under this chapter for the fields of accreditation corresponding to the submitted or generated data, unless this chapter or a covered program exempts a test from requiring accreditation.

(2) The department may not accept data from a laboratory that is not properly accredited under this chapter, except as provided in s. NR 149.11.

(3) The department may initiate enforcement action against a laboratory which maintains accreditations under this chapter but performs testing for analytes for which it does not hold the appropriate accreditation and for which the department offers accreditation when that data will be reported to the department.

(4) A laboratory may not transfer its accreditation to any other entity unless the department expressly approves the transfer by the process specified in s. NR 149.14 (1) (a).

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.06 Certificates. (1) The department shall issue certificates to accredited laboratories indicating or referring to the specific fields of accreditation for which laboratories have been granted accreditation. The department shall issue certificates annually, whenever the fields for which a laboratory is accredited change, and when a laboratory relocates or changes its name.

(2) (ag) The department shall issue certificates to the owner or legally responsible party of a laboratory.

(ar) The department may not issue certificates to anyone who is not the owner or legally responsible party of a laboratory.

(b) The department may indicate in a certificate that a laboratory is managed by an outside contractor.

(3) Certificates are the property of the department and shall be returned to the department upon request.

(4) A laboratory may not alter or modify certificates issued by the department. A laboratory that alters or modifies a certificate, or that misrepresents the fields of accreditation contained or referenced in a certificate, may be subject to revocation of all its accreditations.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; renum. (2) (intro.), (a) to (2) (ag), (ar) under s. 13.92 (4) (b) 1., Stats., Register February 2021 No. 782.

NR 149.08 Acceptance of other accreditations, licenses, or approvals. (1) AGRICULTURE, TRADE, AND CONSUMER PROTECTION AGREEMENT. The department shall accept the accreditation, licensure, or approval by the department of agriculture, trade, and consumer protection for microbiological testing performed by a laboratory submitting or generating data for a covered program.

(2) LABORATORIES ACCREDITED, LICENSED, OR APPROVED BY OTHER GOVERNMENTS. (a) The department may negotiate with and attempt to enter into agreements with federal agencies and agencies of other states to reciprocally accept accreditations of laboratories under this chapter.

(b) The department may accept the accreditation, licensure, or approval of a laboratory by another state or an agency of the federal government if the standards used for the qualification of a laboratory are substantially equivalent to those established in this chapter.

(c) The department may not accept the accreditation, licensure, or approval of a laboratory by another state or an agency of the federal government, unless that state or federal agency accepts laboratories accredited under this chapter.

(3) PRIVATE ORGANIZATION AGREEMENTS. (a) The department may enter into agreements with private not-for-profit organizations to accept accreditation of laboratories under this chapter.

(b) The department may accept the accreditation, licensure, or approval of a laboratory by a private not-for-profit organization if the organization's standards used for the qualification of a laboratory are substantially equivalent to those established in this chapter.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.09 Certification standards review council.

(1) The council shall advise the department on the standards used to certify, register, suspend, and revoke laboratories.

(2) The council shall advise the department on training and outreach activities that the department may offer or sponsor to facilitate compliance of laboratories with this chapter.

(3) The council shall advise the department on the frequency and scope of evaluations necessary to determine compliance of laboratories with this chapter.

(4) The department shall seek the advice of the council before requiring the analysis of additional PT samples and approving PT sample providers.

(5) The department shall seek the advice of the council before implementing changes in the fees assessed to laboratories.

(6) The department shall seek the advice of the council in granting variances.

(7) The department shall prepare annually the following for review by the council:

(a) A summary of laboratory evaluations performed.

(b) A list of required PT samples and available PT sample providers.

(c) A summary of fees scheduled to be assessed to laboratories.

(d) A summary of variances issued.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.10 Enforcement. (1) ADMINISTRATIVE PROCEDURES. A laboratory's accreditation is valid until it expires, is suspended, or is revoked. If, after opportunity for a contested case hearing, the department finds that an accredited laboratory materially and consistently failed to comply with the provisions of this chapter, the department may suspend or revoke a laboratory's accreditation in whole or in part by matrix, analytical technology, method, analyte, or analyte group. Contested case hearings for out-of-state laboratories regulated under this chapter shall be held in Madison, Wisconsin.

(2) SUSPENSION OR REVOCATION OF CERTIFIED LABORATORIES. (a) Causes for suspension of certification include any of the following:

1. Material and consistent failure to comply with the requirements of this chapter.

2. Reporting data to the department after a laboratory is deemed temporarily incapable of performing analysis in any matrix, analytical technology, method, analyte, or analyte group.

3. Suspension of certification, accreditation, license, or approval by another state or agency of the federal government for which the laboratory holds certification if the grounds for suspension are substantially equivalent to any of those listed in this paragraph.

(b) Causes for revocation of certification include any of the following:

1. Material and consistent failure to maintain records as required in this chapter.

2. Failure to allow the department to perform on-site evaluations as specified in subch. VI.

3. Material and consistent failure to comply with the requirements of this chapter.

4. Material and consistent failure to submit requested records to the department.

5. Material and consistent failure to follow specified procedural or quality control requirements prescribed in methods.

6. Falsification of analytical results, testing dates, or any other information submitted to the department by the laboratory. Falsification includes alteration or modification of a certificate.

7. Failure of two consecutive PT samples for any method and analyte or analyte group combination for laboratories holding certification in the drinking water matrix.

8. Revocation of certification, registration, accreditation, license, or approval by another state or agency of the federal government for which the laboratory holds certification if the grounds for revocation are substantially equivalent to any of those listed in this paragraph.

(3) REVOCATION OF REGISTERED LABORATORIES. Causes for revocation of registration include any of the following:

(a) Material and consistent failure to maintain records as required in this chapter.

(b) Failure to allow the department to perform on-site evaluations as specified in subch. VI.

(c) Material and consistent failure to comply with the requirements of this chapter.

(d) Material and consistent failure to submit requested records to the department.

(e) Material and consistent failure to follow specified procedural or quality control requirements prescribed in approved methods.

(f) Falsification of analytical results, testing dates, or any other information submitted to the department by the laboratory. Falsification includes alteration or modification of a certificate.

(4) PROCEDURE FOR SUSPENSION OR REVOCATION OF ACCREDITATION. (a) An order suspending or revoking accreditation shall be mailed to the laboratory and shall state the reasons for suspension or revocation. The order shall include the conditions under which reapplication will be accepted. For orders suspending accreditation, the order may include a timetable for correcting the deficiencies that led to the suspension. For orders revoking accreditation, the department may set a time for the revocation.

(b) An order suspending or revoking an accreditation shall take effect on the 30th day after the order is mailed unless the owner of an accredited laboratory submits a petition for a hearing. Petitions for a hearing shall be submitted to the department within 30 days of receiving the order. The petition for hearing shall specify the findings or conclusions, or both, that the laboratory disputes and conform to the requirements of s. NR 2.05 (5).

(c) If a request for a hearing is submitted and meets the requirements of s. 227.42, Stats., the suspension or revocation shall be stayed, and the department shall conduct a contested case hearing on the matter. At least ten days prior to the date of the hearing, the department shall send a written notice to the laboratory indicating the date, time, and location of the hearing. The final determination of the department, including the basis for the decision, shall be provided by written order to the laboratory after the hearing.

Note: Refer to ch. NR 2 for additional information on the contested hearing process.

(d) The final determination of the department is subject to review under ch. 227, Stats.

(5) REAPPLICATION FOLLOWING SUSPENSION OR REVOCATION. (a) A laboratory that has had its accreditation suspended may reapply for accreditation if all the following are met:

1. The deficiencies that led to the suspension have been corrected in accordance with the timetable contained in the order.

2. Any conditions for reapplication specified in the order have been met.

(b) A laboratory that has had its accreditation revoked may reapply for accreditation if all of the following have been met:

1. The deficiencies that led to the revocation have been corrected.

2. Conditions contained in the order have been satisfied.

3. The time for which the revocation is in effect has expired.

(c) Laboratories reapplying for accreditation following suspension or revocation shall submit an initial application as identified in s. NR 149.14 (1) and (2).

(6) REFERRAL. (a) Any violation of this chapter may be referred to the attorney general's office for enforcement under ss. 299.95 and 299.97, Stats.

(b) Any laboratory operating without proper accreditation for which analysis results are submitted to the department for compliance monitoring or for analyses that require certification or registration under any covered program may be referred by the department to the attorney general's office for enforcement.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.11 Discretionary acceptance. **(1)** Except for results of tests required under ch. NR 809 the department may accept, on a case-by-case basis, the results of tests originating in a laboratory not accredited as required by a covered program if the results meet all other requirements of this chapter.

(2) The requirements of this chapter may be waived by the department when there is a multi-agency response to a hazardous substance discharged in boundary areas of the state.

(3) The requirements of this chapter may be waived by the department when the environmental protection agency national enforcement investigations center laboratory is utilized for EPA or department led enforcement cases.

(4) The department may not accept the results of tests originating in a laboratory not accredited, unless the results are generated in accordance with requirements substantially equivalent to those outlined in this chapter.

Note: Refer to s. NR 149.42 for additional information on the use of alternative methods.

(5) The department may charge a fee under s. 299.11 (5) (d), Stats., if it is necessary to verify the results of tests for which a laboratory requests discretionary acceptance.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.12 Variances. **(1) GENERAL.** The department may approve variances from non-statutory requirements of this chapter when the department determines that the variances have no effect on the department's objectives. Before granting variances, the department shall consider factors such as good cause, circumstances beyond the control of the laboratory, and financial hardship.

(2) REQUEST FOR VARIANCE. Requests for variances shall be submitted to the department. Each variance request shall contain all the following:

(a) The name of the applicant or laboratory.

(b) The section of this chapter from which a variance is sought.

(c) A description of the circumstances under which the variance will be exercised, including any pertinent background information relevant to making a justification.

(3) APPROVAL OF VARIANCE. The department shall approve or deny the requested variance to the applicant within 60 days of receiving all the information referenced in sub. (2). If the request is denied, the department shall state the reasons for the denial.

(4) REPEAL OF VARIANCES. The department will annually review approved variances and may repeal those where the initial

justification for the variance no longer applies. Once the department notifies the laboratory of the repeal, the laboratory will have six months before the repeal is effective.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

Subchapter III — Program Structure

NR 149.13 Fields of accreditation: certification and registration. (1) **GENERAL.** The department shall certify and register laboratories by specific fields of accreditation. Accreditation shall be by certification under s. 299.11 (7), Stats., or registration under s. 299.11 (8), Stats. Fields of accreditation consist of 3 tiers describing the analytical capability of laboratories.

(2) **TIER 1 – MATRIX.** The first tier of accreditation is comprised of aqueous, non-aqueous, and drinking water matrices.

Note: Biosolids and sludges are a non-aqueous matrix for accreditation purposes.

(3) **TIER 2 – TECHNOLOGY OR METHOD.** (ag) The second tier of accreditation is comprised of analytical technologies for the aqueous and non-aqueous matrices or methods for the drinking water matrix.

(ar) The department may certify or register laboratories that analyze aqueous and non-aqueous matrices for the analytical technologies contained in this section, Table 1.

(b) The department shall include any associated sample preparation techniques, such as digestions, distillations, extractions, cleanups, concentrations, and dilutions as part of the certification or registration for a given field of accreditation.

(c) Laboratories may employ multiple methods of analysis for a given analytical technology under the same field of accreditation.

Table 1 - Analytical Technologies for Aqueous and Non-Aqueous Matrices

Number	Analytical Technology
General Chemistry	
1.	Oxygen Demand Assays (BOD or cBOD) ¹
2.	Colorimetric or Turbidimetric
3.	Electrometric Assays (i.e. ion-selective electrode)
4.	Gravimetric Assays – Residue (solids)
5.	Extraction/Gravimetric Assays – Oil & Grease as Hexane Extractable Materials (HEM) ¹
6.	Titrimetric or Potentiometric Titration Assays
7.	Flow Injection-Gas Diffusion–Amperometry ¹
8.	Nondispersive Infrared (NDIR) or Microcoulometry
9.	Ion Chromatography (IC)
Metals	
10.	Flame Atomic Absorption Spectrophotometry (FLAA)
11.	Flame Photometry Spectrophotometry (FP)
12.	Gaseous Hydride Atomic Absorption Spectrophotometry (GHAA)
13.	Graphite Furnace Atomic Absorption Spectrophotometry (GFAA)
14.	Cold Vapor Atomic Absorption Spectrophotometry (CVAA)
15.	Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS)
16.	Thermal Decomposition Atomic Absorption Spectrophotometry (TDAA)
17.	Inductively Coupled Plasma Emission Spectrophotometry (ICP)
18.	Inductively Coupled Plasma-Mass Spectrometry (ICP/MS)
Organics	
19.	Gas Chromatography (GC)
20.	Gas Chromatography-Mass Spectrometry (GC/MS)
21.	Liquid Chromatography (LC)
22.	Liquid Chromatography-Mass Spectrometry (LC/MS)
23.	High Resolution Gas Chromatography-Mass Spectrometry (HRGC/MS)
Other	
24.	Hazardous Waste Characteristics ²
25.	Solid Waste Leaching Procedures ²
26.	Whole Effluent Toxicity Assays ¹
27.	Other ³

1. Accreditation for this technology is only available for the aqueous matrix – Tier 1.

2. Accreditation for this technology is only available for the non-aqueous matrix – Tier 1.

3. The department may offer accreditation in other analytical technologies if the technology is approved by the EPA or is approved by the department as an emerging technology.

(d) The department may certify laboratories analyzing drinking water samples using methods promulgated or approved by the EPA under 40 CFR Part 141.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(4) **TIER 3 – ANALYTES OR ANALYTE GROUP.** (ag) The third tier of the accreditation is comprised of analytes or analyte groups. The department may certify or register laboratories by analyte groups if it improves the efficiency of administering accreditations.

(ar) The analytes and analyte groups available for accreditation under this subsection are contained in Appendix I.

(b) The department, upon consultation with the council, may offer accreditation for additional analytes or analyte groups that are not contained in Appendix I upon request by the manager of a covered program or when the EPA requires the additional analytes or analyte group analysis.

(c) For aqueous and non-aqueous matrices, the analytes and analyte groups are organized into classes. Laboratories analyzing aqueous and non-aqueous matrices may be accredited for analytes and analyte groups belonging to the analytical classes contained in this section, Table 2.

Table 2 - Analytical Classes for Aqueous and Non-Aqueous Matrices

Number	Analytical Classes
1.	General Chemistry
2.	Metals
3.	Volatile Organic Compounds
4.	Base, Neutral, and Acid Extractable Semivolatile Compounds, including: a. Aldehydes and Ketones b. Benzidines c. Chlorinated Hydrocarbons d. Explosive Residues e. Haloethers f. Nitroaromatics g. Nitrosamines h. Non-halogenated Organics i. Phenols j. Phthalates k. Polynuclear Aromatic Hydrocarbons
5.	Polynuclear Aromatic Hydrocarbons
6.	Pesticides and Metabolites, including: a. Acid b. Nitrogen c. Carbamate d. Organochlorine e. Organophosphorus f. Triazine g. Other
7.	Persistent Organic Pollutants
8.	Hazardous Waste Characteristics ¹
9.	Leaching Procedures ^{1,2}
10.	Solvent Scans
11.	Toxicity, Acute
12.	Toxicity, Chronic

1. Hazardous Waste Characteristics and Leaching Procedures are only offered for non-aqueous matrices – Tier 1.

2. Leaching Procedures require that laboratories also maintain accreditation for any analyte to be determined in the resulting leachate in the non-aqueous matrix.

(d) For the drinking water matrix, the analytes and analyte groups are organized into classes. Laboratories analyzing drinking water may be certified for analytes or analyte groups belonging to the analytical classes contained in this section, Table 3.

Table 3 - Analytical Classes for the Drinking Water Matrix

Number	Analytical Classes
1.	Disinfection By-products
2.	Primary Inorganic Contaminants (Non-Metals)
3.	Primary Inorganic Contaminants (Metals)
4.	Secondary Contaminants (Non-Metals)
5.	Secondary Contaminants (Metals)
6.	Synthetic Organic Contaminants (SOC) – Dioxin
7.	Synthetic Organic Contaminants (SOC) – Organochlorine Pesticides
8.	Synthetic Organic Contaminants (SOC) – Nitrogen-Phosphorus Pesticides
9.	Synthetic Organic Contaminants (SOC) – Herbicides
10.	Synthetic Organic Contaminants (SOC) – Miscellaneous
11.	Volatile Organic Compounds (VOC)

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; renum. (3) (intro.), (a), (4) (intro.), (a) to (3) (ag), (ar), (4) (ag), (ar) under s. 13.92 (4) (b) 1., Stats., and correction in (3) (d) made under s. 35.17, Stats., Register February 2021 No. 782.

Subchapter IV — Accreditation Process

NR 149.14 Application for accreditation. (1) GENERAL REQUIREMENTS. (a) Laboratories are required to do all the following:

1. Submit applications to seek, revise, or transfer accreditations.
2. Declare the fields of accreditation being sought, revised, or transferred in corresponding applications.
3. For drinking water, declare the methods of analysis for analytes and analyte groups in the fields of accreditation being sought, revised, or transferred.
4. Submit a current analytical instrument list.
5. Submit acceptable results for PT samples when the department requires the PT sample analysis.
6. For laboratories that are not physically located in Wisconsin, submit a statement of intent to perform analyses for regulatory samples originating in Wisconsin. Intent to perform analyses for regulatory samples originating in Wisconsin can be manifested by any of the following:
 - a. Referencing the affiliation of the applicant laboratory with a plant, office, laboratory, or engineering firm physically located in the state of Wisconsin.
 - b. Submitting a letter from a potential client requesting the applicant to perform analyses to determine compliance with a covered program.
7. Submit any information identified in an application or upon request of the department such as standard operating procedures or analytical data.
8. When the department determines that an evaluation is necessary to determine potential or actual compliance with this chapter, allow the department to perform an on-site evaluation.
9. Remit any necessary fees required under this chapter.
10. Agree to comply with this chapter by signing the application.

(b) The department may not accept applications from a laboratory to which any of the following apply:

1. The laboratory has been issued a notice of violation for nonconformance with this chapter if the nonconformance has not been corrected and the notice of violation has not been closed.
 2. The laboratory has been issued an administrative order of suspension or revocation for a violation of this chapter when the violation has not been closed and the suspension or revocation period specified in an order has not expired.
 3. The laboratory was not in compliance with this chapter at the time the laboratory voluntarily relinquished its accreditations, nonconformances existing prior to relinquishing the accreditations have not been corrected, and at least 6 months have not elapsed since the voluntary action was undertaken.
- (c) The department shall expire any application from a laboratory that has not submitted all the information and materials required as part of the application, or subsequent audit process, within a year of the receipt of the application form.
- (d) The department may require the submittal of additional information necessary, such as standard operating procedures or analytical data, to determine a laboratory's actual or potential compliance with the provisions of this chapter.

(2) INITIAL APPLICATIONS. (a) A laboratory seeking direct accreditation by the department that has never been accredited under this chapter, that has let its entire accreditation lapse for

more than one year, or that has voluntarily relinquished all its accreditations shall submit an initial application to become accredited.

(b) A laboratory seeking reinstatement of its accreditations, following a suspension or revocation, shall submit an initial application for the desired accreditations.

(3) REVISED APPLICATIONS. (a) A laboratory holding valid accreditations shall submit a revised application to seek additional accreditations in any of the following:

1. Matrices.
2. Technologies for an accredited matrix.
3. Analytes or analyte groups within an accredited analytical technology.
4. Analyte-method or analyte group-method combinations for the drinking water matrix.

(b) A laboratory seeking reinstatement of accreditations within a year after failing to renew those accreditations shall submit a revised application for the desired accreditations.

(c) A laboratory seeking to convert a valid certification into a registration or a registration into a certification shall submit a revised application.

(4) APPLICATIONS FOR ACCREDITATIONS THROUGH RECIPROCAL AGREEMENT ACCEPTANCE. (a) A laboratory holding valid accreditations, licenses, or approvals from government bodies or private organizations, with which the department has established a reciprocal agreement, may have its accreditations, licenses, or approvals considered for acceptance by the department by submitting a reciprocity application.

(b) A laboratory applying for acceptance under an existing reciprocal agreement shall do all the following:

1. Submit certificates or official documents of the laboratory's accreditations, licenses, or approvals with its application.
2. Agree to notify the department of any changes, within 30 days of a change in its accreditation, licensure, or approval status with the entity with which the department has the agreement.
3. Submit a copy of the report of the most recent on-site evaluation performed by the entity with which the department has the agreement.

(5) ISSUANCE OF ACCREDITATIONS. (a) The department shall issue accreditations to laboratories through certificates that meet the criteria specified in s. NR 149.06.

(b) The department shall issue a certificate to a laboratory submitting an initial, revised, or reciprocity application for accreditation within 30 days of the date by which the laboratory successfully completes an on-site evaluation or the date by which the department waives an on-site evaluation, subject to all the following:

1. The department may not schedule or waive an on-site evaluation of an applicant laboratory until all the requirements of sub. (1) have been completed.
2. A laboratory completes an on-site evaluation successfully when it addresses, to the department's satisfaction, any deficiencies encountered during the on-site evaluation.

(c) Following an on-site evaluation, the department may issue accreditations, on a case-by-case basis, that are unaffected by any deficiencies encountered during the on-site evaluation.

(d) The department shall issue a revised certificate of accreditation to an accredited laboratory within 30 days of the occurrence of any of the following:

1. Receiving notification from that laboratory that it is changing its name without changing ownership.

2. Approval of relocation to a new facility that does not compromise the laboratory's ability to meet the requirements of this chapter.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (2) (a) made under s. 35.17, Stats., Register February 2021 No. 782.

NR 149.15 Period, renewal, and expiration of accreditation. (1) ACCREDITATION PERIOD. (a) The accreditation period shall commence on September 1 and end on August 31 of the following year for all laboratories accredited by the department.

(b) The department shall renew the accreditations of laboratories that meet the requirements of this section prior to September 1 of each year.

(2) RENEWAL PROCESS. Annually, each laboratory holding valid accreditations under this chapter and wishing to renew its accreditations shall do all the following:

(a) Pay the required annual renewal fee and any assessed administrative fees prior to July 1. After July 1, a late renewal fee may be assessed to laboratories that have not paid all requisite fees. A laboratory is not eligible for renewal of accreditation if full payment is not received prior to September 1.

(b) Submit acceptable PT sample results as required in subch. V, no later than August 31.

(c) If accredited via reciprocal agreement, submit documentation of accreditations and a copy of the most recent on-site evaluation report from the entity with which the department has the agreement.

(3) EXPIRATION OF ACCREDITATIONS. On September 1 of each year, the department shall expire the affected accreditations of laboratories failing to provide the information and fees specified in sub. (2).

(4) VOLUNTARY WITHDRAWAL OF ACCREDITATIONS. Laboratories may voluntarily withdraw accreditations at any time by notifying the department in writing.

Note: Conditions associated with applying for analytes for which accreditation was voluntarily withdrawn are provided in s. NR 149.14 (1) (b) 3.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.155 Required notifications. (1) LABORATORY NAME CHANGE. A laboratory that changes its name without changing ownership shall notify the department, in writing, within 30 days of the effective date of the name change. The department may not charge a fee for any processing resulting solely from a name change.

(2) LABORATORY OWNERSHIP CHANGE. A laboratory that changes its ownership shall notify the department, in writing, within 30 days of the effective date of the ownership change. Notification shall be in the form of a completed application for transfer of ownership.

(3) LABORATORY RELOCATION. A laboratory relocating shall notify the department, in writing, at least 30 days prior to the relocation. Notification shall include the new address and any changes in contact information.

(4) KEY PERSONNEL CHANGES. A laboratory making changes to key personnel, including lab director, lab manager, quality assurance manager, or whole effluent toxicity technical expert, shall notify the department within 30 days of these changes.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.18 Subcontracting. (1) Subcontracting samples shall be to a laboratory that holds valid certifications corresponding to the matrix, technology or method, and analyte requested.

(2) A laboratory accepting samples under a subcontract from another laboratory shall maintain any analytical records needed to determine compliance with this chapter. The records shall be made available to the laboratory providing the samples.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.19 Requirements for certification in the drinking water matrix. (1) APPLICABILITY. This section contains additional requirements that apply to laboratories analyzing drinking water for compliance under ch. NR 809.

(2) GENERAL REQUIREMENTS. (a) The minimum criteria and procedures for certification in the drinking water matrix are specified in the following documents:

1. As updated, the "Manual for the Certification of Laboratories Analyzing Drinking Water," EPA 815-R-05-004, fifth edition, EPA, Office of Ground Water and Drinking Water, January 2005.

2. As updated, the "Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water," EPA 815-F-08-006 EPA, Office of Ground Water and Drinking Water, June 2008.

Note: The documents above can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(b) The department may not grant either interim or provisional certifications.

(c) A laboratory shall follow any additional criteria and procedures identified in this chapter applying to drinking water analyses.

(3) REQUIREMENTS FOR INORGANIC CONTAMINANTS. (a) To receive and maintain certification to conduct analyses of inorganic contaminants, the laboratory shall achieve MDLs no greater than the MDLs specified in 40 CFR 141.23 (a) (4) (i) and 40 CFR 141.89 (a) (1) (iii) for each accredited method.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(b) Each laboratory shall successfully analyze at least one PT sample annually according to criteria specified in 40 CFR 141.23 (k) (3) (ii) or 40 CFR 141.89 (a) (1) (ii) (A) and (B) for each accredited method.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(4) REQUIREMENTS FOR VINYL CHLORIDE. (a) To receive and maintain certification to conduct analyses of vinyl chloride, the laboratory shall achieve a MDL no greater than 0.0002 mg/L for each accredited method.

(b) Each laboratory shall successfully analyze at least one PT sample annually for each accredited method according to criteria specified in 40 CFR 141.24 (f) (17) (ii) (B). Vinyl chloride is evaluated separately from the other regulated volatile organic compounds and certification for the regulated volatile organic compounds requires successful analysis of vinyl chloride in addition to requirements for the other regulated volatile organic compounds.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(5) REQUIREMENTS FOR OTHER VOLATILE ORGANIC COMPOUNDS. (a) To receive and maintain certification to conduct analyses of volatile organic compounds, excluding vinyl chloride, the laboratory shall achieve MDLs no greater than 0.0005 mg/L for all regulated volatile organic compounds for each accredited method.

(b) Each laboratory shall successfully analyze at least one PT sample annually for each accredited method according to criteria specified in 40 CFR 141.24 (f) (17) (i) (B). Excluding vinyl chlo-

ride, a laboratory may be certified for all volatile organic compounds if the laboratory successfully analyzes at least 80% of the regulated volatile organic compounds.

Note: Some PT sample providers include the trihalomethanes in the sample for regulated volatile organic compounds. Trihalomethanes are not considered part of the "80%" rule. To be accredited for the regulated volatile organic compounds, vinyl chloride and 16 of the remaining 20 regulated volatile organic compounds are to pass in each PT sample.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(6) REQUIREMENTS FOR SYNTHETIC ORGANIC CONTAMINANTS. (a) To receive and maintain certification to conduct analyses of synthetic organic contaminants, the laboratory shall achieve MDLs no greater than the MDLs specified in 40 CFR 141.24 (h) (18) for each accredited method.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(b) Each laboratory shall successfully analyze at least one PT sample annually according to criteria specified in 40 CFR 141.24 (h) (19) (i) (A) and (B).

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(7) REQUIREMENTS FOR DISINFECTION BY-PRODUCTS. (a) To receive and maintain certification to conduct analyses of disinfection by-products, the laboratory shall meet the requirements specified in 40 CFR 141.131 (b) (2) (iv) for each accredited method. To receive certification to conduct analyses of trihalomethanes, the laboratory shall achieve MDLs no greater than 0.0005 mg/L for each regulated analyte for each accredited method.

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(b) For the haloacetic acid and trihalomethane PT samples, laboratories shall pass 80%, or 4 of the analytes present in each PT sample.

(8) FAILED PT SAMPLES. The department may not renew the accreditation for analytes for which the laboratory fails consecutive PT samples.

(9) CERTIFICATION EXEMPTIONS. Certification is not required to perform any of the following analyses:

- (a) Fluoride analysis required under s. NR 809.74.
- (b) Analysis for free chlorine residual and total chlorine residual required under s. NR 809.74.
- (c) Analysis for pH required under s. NR 809.548.
- (d) Analysis for turbidity required under s. NR 809.113.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; (1) (title), (8) (title) created under s. 13.92 (4) (4) (b) 2., Stats., Register February 2021 No. 782.

NR 149.20 Requirements for whole effluent toxicity testing. All the following apply to laboratories accredited to perform whole effluent toxicity testing:

(1) ACUTE AND CHRONIC WHOLE EFFLUENT TOXICITY TESTING BY SPECIES. Laboratories analyzing whole effluents for acute and chronic toxicity for a given species shall follow the quality control requirements referenced in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual," as updated.

Note: Links to the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual," can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(2) ACCREDITATION REQUIREMENTS FOR CHEMICAL TESTING IN SUPPORT OF WHOLE EFFLUENT TOXICITY TESTING. Water chemistry testing performed in support of whole effluent toxicity testing for ammonia, alkalinity, hardness, pH, dissolved oxygen,

conductivity, and total residual chlorine do not require separate accreditation under this chapter.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.21 Fees. The department shall establish fees for laboratories participating in the laboratory accreditation program. These costs include those associated with laboratory evaluations, discretionary acceptance of data, reciprocity, training, and collection of fees. Fees may not be prorated and, except for overpayment, are not refundable.

(1) TOTAL FEE INCOME. (a) The laboratory accreditation program's total fee income shall be designed to generate revenues equal to the costs of administering this chapter. Any amendments to the formulas in this subsection shall be reviewed by the council prior to being proposed as rule amendments.

(b) The department may adjust the fee schedule according to the formulas in this subsection and the relative value unit items specified in Tables 1, 2, and 3. Annual fee adjustments shall be reviewed by the council and approved annually by the natural resources board.

(c) The following formulas shall be used to generate and adjust the laboratory accreditation program's fee schedule:

1. Fee Revenue Required = Projected Laboratory Accreditation Program Expenses - (Application Fees + Travel Reimbursement)

a. Fee Revenue Required is the total amount of revenue which shall be collected via fees to cover all laboratory accreditation program costs.

b. "Laboratory Accreditation Program Expenses" is the sum of all anticipated laboratory accreditation program expenses including salary, fringes, evaluation travel costs, supplies, and services. This includes travel costs for evaluation of out-of-state labs which are required to reimburse the laboratory accreditation program for laboratory evaluation travel costs. Application Fee revenues are excluded from the Fee Revenue required because the application fee revenues are variable and collected independently throughout an accreditation period.

Note: "Laboratory accreditation program expenses" may not exceed the legislature's approved spending authority for the laboratory accreditation program in a fiscal year. The department of administration approved spending authority is given in s. 20.370 (9) (fj), Stats., and may be revised by the department of administration to recover laboratory accreditation program cost.

c. Application Fees is a three-year moving average of application fees received for the three most recent fiscal years.

d. Travel Reimbursement is a three-year moving average of out-of-state travel reimbursements for the three most recent fiscal years. Laboratory accreditation program costs related to travel for out-of-state audits are negated because the department recovers these costs directly from each lab.

Note: For example, given the following,

Projected Laboratory Accreditation Program Expenses = \$612,121

Application Fees (three-year average) = \$31,681

Travel Reimbursement (three-year average) = \$17,079

Fee Revenue Required would be \$612,121 - (\$31,681 + \$17,079) = \$563,361.

2. Total # RV Units = $\sum [(\# \text{Laboratories in item}) \times (\text{RV of item})]$.

a. Total # RV Units is the total number of RV units available for the fiscal year. It is the mechanism by which fees are distributed to individual laboratories.

b. "# Laboratories in item" is a count of how many laboratories will be assessed the fee for that item for a fiscal year, based on accreditations currently held.

c. The RV units for each fee item, "RV of item," are listed in Table 3. The total number of RV units is the sum of all base fee RV, matrix fee RV, and technology or class fee RV.

Note: For example, given the following,

Base RV: # Labs Registered (5 RV) = 225; # Labs Certified (10 RV) = 141;

Matrix RV = 341 Aqueous, 71 Solid, and 43 Drinking water.
 Technology/Class RV= 2612 Aqueous, 943 Solid, and 575 Drinking water.
 Base Fee RV Units = (225 x 5) + (141 x 10) = 2535
 Matrix RV Units = (341+ 71+43) = 455 x 5 RV/Matrix = 2275
 Technology/Class RV Units = (2612+ 943+575) = 4130 RV
 Total # RV Units = 2535 + 2275 + 4130 = 8940 RV Units.

3. Cost per RV = Fee Revenue Required / Total # RV Units.
 The Cost per RV is the dollar value assigned to one RV unit and is used to establish all fees for items in Table 3 of this section. The cost per RV is rounded to the nearest \$0.50 to simplify fee statements.

Note: For example, given the following,
 Fee Revenue Required = \$563,361
 Total # RV Units = 8940 RVU
 Cost per RV (\$/RV) = \$563,361 / 8940 RVU = \$63.01/RV Units; rounded to the nearest \$0.50 = \$63.00/RV Units.

4. Laboratory fees = (# RV units for a given laboratory) x (Cost per RV). The sum of base, matrix, technology, and class fees for a given laboratory is multiplied by the cost per RV to determine the fee for each laboratory. Any outstanding administrative fees may also be added.

Note: For example, given the following for Pinestump Wastewater Treatment Plant Laboratory,
 Base RV: 5 (registered)
 Matrix RV =5 (aqueous matrix only)
 Technology/Class RV= 4 (Oxygen Demand Assays = 3 RV; Gravimetric Assays - Residue = 1 RVU).
 Total # RV Units = 5 + 5 + 4 = 14 RV Units
 Laboratory fee = 14 RV Units x \$63.00/RV Units = \$882.00.

(2) ADMINISTRATIVE FEES. The department shall assess fees to recover the cost of specified administrative functions specified in this section, Table 1. Any outstanding administrative fees may be included as part of the annual fee.

Table 1 - Administrative Fees

Item	RV Units
Discretionary Acceptance (s. NR 149.11)	Actual Cost
Evaluation Cancellation ¹	Incurred Costs
Evaluation for Enforcement Follow-Up	Actual Cost
Evaluation of Out-of-State Laboratories	Travel Cost
Late Renewal Fee ²	2

¹ Out-of-state laboratories may be required to reimburse the laboratory accreditation program for travel costs incurred by the cancellation or postponement of an evaluation, including airfare, hotel, and rental car expenses.

² Assessed 30 days after payment due date.

(3) APPLICATION FEES. (a) The department shall assess fees for all applications specified in this section, Table 2.

(b) The fee for an application also includes matrix and technology or class fees when a laboratory applies for a new matrix, technology, or class. The matrix fee is not required if a laboratory is applying for additional technologies or analytes within a matrix for which the lab already holds accreditation. Technology fees are not required if a laboratory already holds accreditation for that matrix and technology or matrix and class, for drinking water combination.

Note: Example – The application fee for a laboratory applying to add ammonia by colorimetry under the aqueous matrix is based on only the number of RV units for a revised application since the lab has already paid for the aqueous matrix and colorimetry technology as part of its renewal fees.

(c) Application fees are not refundable in either whole or part.

(d) If an application is not completed within a single fiscal year, the department may adjust the fees on the application to recover the difference in fees between the year the application was submitted and the year the application was completed. The laboratory shall pay this difference prior to receiving accreditations.

Table 2 - Application Fees

Item	RV Units
Initial Application	6
Revised Application	3
Reciprocity Application	4
Transfer of Ownership Application	4

(4) ANNUAL FEES. The department shall assess an annual fee to each laboratory holding accreditations under this chapter either directly or through agreements. A laboratory's annual fee shall be the sum of all the following:

(a) The base fees for accreditation. The department shall assess a base fee to all laboratories holding accreditations under this chapter. The number of RV units assigned to each type of base fee is specified in Table 3 of this subchapter.

(b) The matrix fees. The department shall assess a fee per matrix type to all accredited laboratories. The number of RV units assigned to each type of matrix fee is specified in this section, Table 3.

(c) All of the following analytical fees:

1. Analytical technology fees. The department shall assess a fee for each analytical technology, per matrix, to all accredited laboratories, in fields involving the aqueous and non-aqueous matrices. The assessed fee shall be based on the RV units specified in this section, Table 3.

2. Analytical class fees. The department shall assess a fee, per analytical class, to all certified laboratories in fields involving the drinking water matrix. The assessed fee shall be based on the RV units specified in this section, Table 3.

(d) Any outstanding administrative fees.

Table 3 - Annual Fees for Accreditation

	Item	RV Units
A.	Administrative Fees	
	Outstanding administrative fees	per Table 1 of this subchapter
B.	Base Fees	
	Base Fee, Certification	10
	Base Fee, Registration	5

	Item	RV Units
C.	Matrix Fees	
	Matrix Fee, Aqueous	5
	Matrix Fee, Drinking Water	5
	Matrix Fee, Non-Aqueous	5
D.	Analytical Technology Fees for Aqueous and Non-Aqueous Matrices	
	General Chemistry	
	Oxygen Demand Assays (BOD or cBOD)	3
	Colorimetric or Turbidimetric	2
	Electrometric Assays (i.e. ion-selective electrodes)	1
	Gravimetric Assays - Residues (solids)	1
	Extraction/Gravimetric Assays - Oil & Grease as Hexane Extractable Materials (HEM)	2
	Titrimetric or Potentiometric Titration Assays	1
	Flow Injection–Gas Diffusion–Amperometry	4
	Nondispersive Infrared (NDIR) or Microcoulometry	2
	Ion Chromatography (IC)	4
	Metals	
	Flame Atomic Absorption Spectrophotometry (FLAA)	2
	Flame Photometry Spectrophotometry (FP)	2
	Gaseous Hydride Atomic Absorption Spectrophotometry (GHAA)	3
	Graphite Furnace Atomic Absorption Spectrophotometry (GFAA)	3
	Cold Vapor Atomic Absorption Spectrophotometry (CVAA)	3
	Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS)	3
	Thermal Decomposition Atomic Absorption Spectrophotometry (TDAA)	3
	Inductively Coupled Plasma Emission Spectrophotometry (ICP)	4
	Inductively Coupled Plasma-Mass Spectrometry (ICP/MS)	5
	Organics	
	Gas Chromatography (GC)	4
	Gas Chromatography-Mass Spectrometry (GC/MS)	5
	Liquid Chromatography (LC)	4
	Liquid Chromatography-Mass Spectrometry (LC/MS)	5
	High Resolution Gas Chromatography-Mass Spectrometry (HRGC/MS)	10
	Other	
	Hazardous Waste Characteristics	2
	Solid Waste Leaching Procedures	2
Whole Effluent Toxicity Assays	5	
Other	Not to exceed 10 ¹	
E.	Analytical Class Fees for Drinking Water Matrix	
	Disinfection By-products	5
	Primary Inorganic Contaminants (Non-Metals)	3
	Primary Inorganic Contaminants (Metals)	6
	Secondary Contaminants (Non-Metals)	2
	Secondary Contaminants (Metals)	3
	Synthetic Organic Contaminants (SOC)– Dioxin	8
	Synthetic Organic Contaminants (SOC)– Organochlorine Pesticides	3
	Synthetic Organic Contaminants (SOC)– Nitrogen-Phosphorus Pesticides	3
	Synthetic Organic Contaminants (SOC)– Herbicides	3
	Synthetic Organic Contaminants (SOC)– Miscellaneous	4
	Volatile Organic Compounds (VOC)	4
	Other	Not to exceed 10 ¹

¹ Actual cost will be determined by the department considering the complexity of the technology.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (4) (c) (intro.) made under s. 35.17, Stats., Register February 2021 No. 782.

Subchapter V — Proficiency Testing

NR 149.22 Required proficiency testing samples and frequency of analysis. (1) REQUIREMENTS. (a) A laboratory shall participate in at least one PT sample study per accreditation period as specified in sub. (2), subject to all the following:

1. For aqueous and non-aqueous matrices, a laboratory shall analyze aqueous matrix PT samples for each combination of technology and analyte or analyte group in its fields of accreditation.
2. For the drinking water matrix, a laboratory shall analyze PT samples for each combination of method and analyte or ana-

lyte group in its fields of certification. Acceptance criteria for these samples are set in s. NR 149.27.

(b) PT samples may be those offered by approved PT sample providers at regular intervals, as “rapid response” PT samples, or as custom formulations approved by the department.

(c) A laboratory shall report a proper method code, which matches the technology and analyte or analyte group for which accreditation is held, with results for PT samples.

Note: A link to the universal list of method codes for methods and technologies is available from the NELAC Institute (TNI) which can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(2) LISTS OF REQUIRED PT SAMPLES AND APPROVED PT SAMPLE PROVIDERS. (a) The department shall seek the advice of the council prior to identifying required PT samples and approved PT sample providers.

(b) The list shall identify matrix-specific PT samples required for submittal for renewal of accreditation or with initial or revised applications and the specific PT sample providers approved for supplying each required PT sample.

Note: Lists of required PT samples and approved PT sample providers can be found on the Wisconsin department of natural resources laboratory accreditation program website.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.23 Approval of proficiency testing sample providers. **(1) CRITERIA FOR APPROVAL.** When evaluating a PT sample provider for approval, the department shall consider criteria including all the following:

(a) The PT sample provider accreditation status by national accreditation programs.

(b) The PT sample provider use of techniques for calculating acceptance limits as specified in s. NR 149.27.

(2) PROFICIENCY TESTING SAMPLE PROVIDER REQUIREMENTS. Approved PT sample providers shall submit all PT sample results to the department electronically in a format specified by the department.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; renumbering in (1) under s. 13.92 (4) (b) 1., Stats., Register February 2021 No. 782.

NR 149.24 Proficiency testing samples for applications and accreditation renewal. **(1) PT SAMPLE ACCEPTANCE CRITERIA.** The department may not grant or renew an accreditation unless the associated PT sample results meet the criteria specified in s. NR 149.27.

(2) PT SAMPLE STUDY CLOSE DATE. Acceptable PT samples shall have a PT sample study close date no more than six months prior to the date of application.

(3) PT SAMPLE DUE DATE FOR RENEWAL. For renewal of accreditations, which begin on September 1 of each calendar year, acceptable PT sample results shall have been reported electronically to the department by an approved PT sample provider no sooner than January 1 or later than August 31 of the same calendar year. Preliminary reports from approved PT sample providers may not be used for renewal of accreditation.

Note: For example, to renew accreditation for any analyte effective for the period from September 1, 2009 to August 31, 2010, a laboratory shall have successfully analyzed a PT sample for that analyte reported between January 1 and August 31, 2009.

(4) PT SAMPLES FOR APPLICATIONS. A laboratory submitting initial or revised applications for accreditation shall analyze PT samples from an approved PT sample provider and submit acceptable results for any of the following:

(a) For aqueous and non-aqueous matrices, acceptable PT sample results are required for each combination of technology and analyte or analyte group for which the department has identified that PT samples are required.

(b) For the drinking water matrix, acceptable PT sample results, from a water supply study, are required for each combination of method and analyte or analyte group.

(5) PT SAMPLES FOR RENEWAL. A laboratory wishing to renew its accreditation shall analyze PT samples from an approved PT sample provider and submit acceptable results for any of the following:

(a) For aqueous and non-aqueous matrices, acceptable PT sample results from a water pollution study are required for each combination of technology and analyte or analyte group for which the department has identified that PT samples are required.

(b) For the drinking water matrix, acceptable PT sample results from a water supply study are required for each combination of method and analyte or analyte group.

Note: The department does not accept PT samples prepared in a non-aqueous matrix to obtain or renew accreditation for analytes or analyte groups under the non-aqueous matrix.

(6) RENEWAL REQUIREMENTS FOR MULTIPLE SUCCESSIVE PT SAMPLE FAILURES. (am) A laboratory that experiences multiple successive PT sample failures shall submit two consecutive acceptable PT samples from an approved PT sample provider to renew its accreditation. Consecutive PT samples shall be two unique studies received by the laboratory at least ten business days apart. The laboratory may not prepare or analyze the two PT samples in the same batch.

(bm) 1. For aqueous and non-aqueous matrices, multiple successive PT sample failure means failing three consecutive PT samples for any combination of technology and analyte or analyte group.

2. For the drinking water matrix, multiple successive PT sample failure means failing two consecutive PT samples for any combination of method and analyte or analyte group.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in numbering in (6) made under s. 13.92 (4) (b) 1., Stats., and correction in (6) (bm) made under s. 35.17, Stats., Register February 2021 No. 782.

NR 149.25 Treatment of proficiency testing samples. **(1)** PT samples shall be subjected to any preparatory steps undergone by analytical samples of that matrix, unless the preparation instructions submitted by a PT sample provider specifically instruct omitting a preparatory step.

Note: Preparatory steps include digestions, distillations, extractions, concentrations, and dilutions.

(2) A laboratory may report multiple results for a single PT sample when the laboratory maintains accreditations for multiple technologies for any analyte or analyte group in aqueous and non-aqueous matrices.

(3) A laboratory may report multiple results of a single PT sample when the laboratory maintains certifications for multiple methods for any analyte or analyte group in the drinking water matrix.

(4) Prior to submitting PT sample results to a PT sample provider, all the following apply:

(a) A laboratory may not send a PT sample, or portion of a PT sample, to another laboratory for analysis.

(b) A laboratory may not knowingly analyze a PT sample, or a portion of a PT sample, from another laboratory.

(c) Until a PT sample study has been closed, a laboratory may not share results of a PT sample from that study to any party other than the PT sample provider or regulatory agency.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.26 Reporting proficiency testing sample results. **(1)** A laboratory shall submit PT sample results to PT

sample providers in accordance with the dates specified by the PT sample providers.

(2) PT sample reports may be submitted to the department directly from the PT sample provider or by the laboratory, but it is the laboratory's responsibility to ensure the department receives the necessary reports for initial and revised applications. The laboratory shall submit PT sample reports in their entirety, without modification, to the department.

(3) Results from all PT sample reports issued to the department by PT sample providers shall be used to determine a laboratory's accreditation status.

(4) The department may only accept amended and reissued PT sample reports if the reissue is due to an error made by the PT sample provider and revised reports are all the following:

- (a) Clearly labeled as revised or reissued.
- (b) Directly submitted to the department by the PT sample provider.
- (c) Accompanied by an explanation of the PT sample provider's error.

Note: Re-issued reports are acceptable in cases when the laboratory neglected to instruct the PT sample provider to report results to the department.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.27 Proficiency testing sample acceptance limits and grading. (1) ACCEPTANCE LIMITS. A laboratory's result for any analyte or analyte group is considered unacceptable if it meets any of the following conditions:

- (a) The result falls outside the acceptance limits.
- (b) The laboratory reports a result for an analyte not present in the PT sample.
- (c) The laboratory does not report a result for an analyte present in the PT sample.
- (d) The laboratory fails to submit its results to the PT sample provider on or before the deadline for the PT sample study.
- (e) The laboratory reports a method code for either an unapproved method or the method code reported is not appropriate for the technology-analyte or method-analyte combination.
- (f) The laboratory fails to meet department specified grading criteria for multi-analyte PT samples.

Note: Department grading criteria can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(2) GRADING. (a) PT samples for analytes in aqueous and non-aqueous matrices shall be graded in accordance with acceptance limits established by the department considering criteria developed by the EPA.

(b) When the EPA has not developed acceptance limits for required PT sample analytes, the department may develop acceptance limits based on its experience or information supplied by approved PT sample providers.

(c) When an insufficient number of laboratories participate in a study to generate peer-based acceptance limits in a PT sample with analytes for which the EPA has not established acceptance limits, the department may grade results using fixed acceptance limits.

(d) PT sample analytes in drinking water shall be graded in accordance with the acceptance limits established in 40 CFR 141.23 (k) (3) (ii), 40 CFR 141.24 (f) (17) (i) (C) and (D), 40 CFR 141.24 (f) (17) (ii) (B), 40 CFR 141.24 (h) (19) (i) (A) and (B), and 40 CFR 141.89 (a) (1) (ii), and 40 CFR 141.131 (b) (2) (ii) and (iii).

Note: Links to 40 CFR Part 141 can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(e) When accreditation in an analyte group is based on passing a representative PT sample containing more than one analyte, the laboratory shall report acceptable results on at least 80% of the analytes to achieve acceptable results for that sample. The department may investigate repeated failures for specific analytes and direct enforcement action in the event of two consecutive failures in the drinking water matrix or three consecutive failures in the aqueous matrix.

(f) The department shall establish procedures for evaluating false positives and false negatives reported in analyzed PT samples.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.28 Procedure for correcting unacceptable proficiency testing sample results. (1) AQUEOUS AND NON-AQUEOUS MATRICES.

(a) If a laboratory does not meet the acceptance limits for an analyte or analyte group and the laboratory does not have acceptable results on a previous sample analyzed during the same accreditation period, the laboratory shall analyze a second PT sample for that analyte or analyte group.

(b) If the results of a second PT sample do not meet the acceptance limits, the department may initiate an assessment of the laboratory's quality control records if this action is necessary to validate data generated by the laboratory. If two consecutive PT samples do not meet acceptance limits, the laboratory shall do all the following:

1. Prepare a corrective action report and initiate an action plan to correct the problems within 30 days of the date of notification of the second failure. This action plan shall include a timetable for correcting the problems and obtaining a third PT sample.

2. Analyze a third PT sample within 60 days of the date of notification of the second failure. If the results of the third PT sample do not meet the acceptance limits, the laboratory shall analyze two subsequent and consecutive acceptable PT samples.

(c) The department may not renew accreditation of those analytes or analyte groups for which a laboratory has failed three consecutive PT samples and has not successfully analyzed two subsequent and consecutive PT samples for those analytes or analyte groups prior to September 1.

(d) When applying to have an analyte or analyte group reinstated after non-renewal for failing three consecutive PT samples, the laboratory shall provide acceptable results on two subsequent and consecutive PT sample studies for that analyte or analyte group. The consecutive PT samples shall be two unique studies received by the laboratory at least ten business days apart. The laboratory may not prepare or analyze the two PT samples in the same batch.

(2) DRINKING WATER. If a certified laboratory does not meet the acceptance limits that have been established by the department, the department shall require the laboratory to analyze a second PT sample and may require the laboratory to submit a corrective action report. If the results of the second sample do not meet the acceptance limits, the department may not renew the laboratory's certification and may revoke the laboratory's certification as specified in s. NR 149.10. To reinstate the certification for the affected method-analyte or analyte group, the laboratory shall submit a revised application, pay the revised application fee, and provide acceptable results on two subsequent and consecutive PT sample studies for that method-analyte or analyte group. The consecutive PT samples shall be two unique studies received by the laboratory at least ten business days apart. The laboratory may not prepare or analyze the two PT samples in the same batch.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

Subchapter VI — On-Site Laboratory Evaluations

NR 149.29 Purpose, type, and frequency. (1) The department shall perform on-site evaluations to determine a laboratory's potential, actual, or continued ability to comply with the provisions of this chapter.

(2) The department shall conduct announced on-site evaluations of laboratories once every three years and when any of the following occurs:

(a) A laboratory applies to become certified or registered in any field of accreditation unless the department waives the requirement to perform an on-site evaluation. When the department does not waive an evaluation, the evaluation shall be performed within 90 days after the department determines that a received application is complete.

(b) A laboratory changes its location, ownership or key personnel, unless the department waives the requirement to perform an on-site evaluation. When the department does not waive an evaluation, the evaluation shall be performed within 90 days after the department receives notification of these changes.

(c) The department determines that an on-site evaluation is necessary to verify corrective action implemented by a laboratory to address deficiencies identified in a previous on-site evaluation.

(d) The department has reason to believe that a laboratory is not in compliance with this chapter.

(3) The department may conduct unannounced on-site evaluations of a laboratory to verify compliance with this chapter after a notice of violation has been issued to a laboratory.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (title) made under s. 13.92 (4) (b) 2., Stats., Register February 2021 No. 782.

NR 149.30 Evaluation procedures and appraisal.

(1) The department shall perform on-site evaluations of laboratories to evaluate systems, practices, procedures, and documentation in a laboratory and to identify deficiencies according to documented procedures that promote consistency in determining a laboratory's potential, actual, or continued ability to comply with this chapter.

(2) If, in performing an on-site evaluation, the department finds that the laboratory is implementing a procedure that is neither allowed nor disallowed by method or this chapter, the department will assess the scientific validity of the procedure. The department may seek the advice of the council in making determinations under this subsection.

(3) The department shall provide laboratories with a survey to allow them to appraise the evaluation process.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (2) made under s. 13.92 (4) (b) 7., Stats., Register February 2021 No. 782.

NR 149.31 Evaluation reports. (1) The department shall document the deficiencies identified during an on-site evaluation under s. NR 149.30 in reports issued to the evaluated laboratory.

(2) The report of an on-site evaluation shall be issued to a laboratory within 30 days of the conclusion of the on-site visit. When the department finds it necessary to issue an evaluation report at a date later than 30 days after the conclusion of an on-site visit, the department shall notify the laboratory about the delay. The notice shall include an expected delivery date for the report.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.32 Evaluation corrective action. (1) A laboratory shall take corrective action to address all deficiencies dis-

covered during an on-site evaluation under s. NR 149.30 and contained in an evaluation report under s. NR 149.31.

(2) A laboratory shall submit to the department, within 30 days from the evaluation report's date, a plan of corrective action to address all the deficiencies noted in the report. When a laboratory finds it necessary to submit a corrective action plan at a date later than 30 days after the evaluation report's date, the laboratory shall notify the department about the delay and provide an expected delivery date in consultation with the department.

(3) The department shall review the corrective action plan submitted by a laboratory under sub. (2) and inform the laboratory whether the submitted plan addresses satisfactorily all noted deficiencies, or whether additional action or documentation is necessary to determine the laboratory's ability to comply with this chapter, subject to all the following:

(a) When the department determines that the submitted corrective action plan addresses all noted deficiencies satisfactorily, the department shall inform the laboratory in writing within 30 days that the plan is acceptable.

(b) When the department determines that additional action or documentation is needed to evaluate compliance with this chapter, the department, in consultation with the laboratory, shall set a date for the laboratory to submit a second corrective action plan.

1. If the department determines that the second corrective action plan submitted under sub. (3) (b) addresses all noted deficiencies satisfactorily, the department shall inform the laboratory in writing that the evaluation process has concluded.

2. If the department determines that the second corrective action plan submitted under sub. (3) (b) does not address all the noted deficiencies satisfactorily, the department may schedule another on-site evaluation to determine the laboratory's compliance with this chapter, terminate any outstanding application that led to the original on-site evaluation, or direct enforcement to the laboratory.

3. If a second on-site evaluation is scheduled as a follow-up to a second corrective action plan submitted under sub. (3) (b), the department shall establish deadlines that resolve any remaining unresolved deficiencies expeditiously, but no later than 90 days after the conclusion of the follow-up visit.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.33 Conflicts of interest. (1) The department shall establish procedures to ensure and document that laboratory evaluators under its employment are free of any conflicts that would render the laboratory evaluator incapable of performing an objective and unbiased evaluation of a laboratory.

(2) A laboratory may request information and documents used by the department to establish that any evaluator assigned to perform the laboratory's evaluation is free of any conflicts of interest.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.34 Evaluator qualifications. (1) The department shall develop procedures to establish and evaluate the education, experience, and credentials of the laboratory evaluators under its employment.

(2) A laboratory may request information and documents used by the department to establish that any evaluator assigned to perform the laboratory's evaluation has the necessary education, experience, or credentials to perform evaluations competently.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

Subchapter VII — Quality Systems

NR 149.35 General requirements. (1) SCOPE. This subchapter establishes personnel, quality assurance, quality control, method selection, sample handling, and documentation requirements for laboratories.

(2) RESPONSIBILITY FOR QUALITY SYSTEM. A laboratory shall conduct analytical activities under a quality system that incorporates the provisions of this subchapter. At least one individual within a laboratory's organization or under the laboratory's employment shall be identified to the department as responsible for establishing, implementing, assessing, and revising, as needed, a laboratory's quality system.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.36 Laboratory personnel. (1) MANAGEMENT AND ANALYTICAL STAFF. The laboratory shall have personnel with education, training, or experience that allows the laboratory to comply with the requirements of this chapter. Contractors, external to the laboratory, may serve in key laboratory roles. When external contractors serve in essential laboratory roles, the contracts shall be available to the department to ensure that contractual specifications satisfy the requirements of this chapter.

Note: For requirements regarding changes in key personnel see s. NR 149.155.

(2) DEMONSTRATION OF CAPABILITY. (a) When a laboratory references a method that contains procedures for demonstrating initial capability, continuing capability or both, personnel performing analyses using these methods shall perform the procedures, meet any associated evaluation criteria, and document the results. When initial demonstrations of capability include the analysis of samples, the samples shall be prepared from a clean matrix and processed through all method preparation steps.

(b) When a laboratory references a method that does not contain procedures for demonstrating initial capability, the laboratory shall establish initial demonstration of capability criteria for determining that each person who performs testing on compliance samples using the method has demonstrated the necessary skills and expertise required to generate quality analytical results. The laboratory shall retain documentation that each person performing a given test on compliance samples has satisfied the demonstration of capability criteria established by the laboratory.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.365 Laboratory ethics. All the following practices are prohibited and may result in enforcement action under s. NR 149.10:

(1) Fabrication, falsification, or misrepresentation of data.

(2) Improper instrument clock setting, termed time traveling, or improper recording of date or time.

(3) Unwarranted manipulation of samples, software, peak integration, or analytical conditions.

(4) Concealing or failing to report a known improper or unethical behavior or action associated with sample analysis.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.37 Quality manual. (1) PURPOSE AND GENERAL PROVISIONS. Each laboratory shall define its quality system in a quality manual. All policies and procedures governing the laboratory's quality system shall be documented or referenced in the quality manual. All laboratory personnel shall follow the policies and procedures established by the quality manual.

(2) FORMAT. The quality manual shall have a format that addresses the content elements specified in this section. Content elements may be presented in narrative, tabular, schematic, or

graphical form. The manual shall be a document in hard copy or electronic format traceable to the laboratory.

(3) CONTENT. Unless included in other standard operating procedures maintained under s. NR 149.40, the quality manual shall include, address, or refer to all the following elements:

(a) Procedures for retention, control, and maintenance of documents used in or associated with analysis.

(b) Procedures for achieving traceability of standards, reagents, and reference materials used to derive any results or measurements.

(c) Procedures for handling samples.

(d) Procedures for calibration, verification, and maintenance of support equipment.

(e) Procedures for evaluating quality control samples.

(f) Procedures for initiating, following up on, and documenting corrective action, addressing quality assurance and quality control failures, and any discrepancies or nonconformances.

(4) REVISIONS. The quality manual shall be kept current. All editions or versions of the quality manual shall indicate the dates in which the quality manual was issued or revised.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.38 Corrective action. (1) The laboratory shall take corrective action in response to any nonconformances including all the following:

(a) Departures from established procedures in the quality system are identified.

(b) Quality control samples fail, unless immediate reanalysis of the affected sample resolves the issue.

(2) The corrective action under sub. (1) shall identify the problem, determine the most probable cause of the problem, implement solutions to correct the problem, and include a mechanism to verify that the action has had the desired effect.

(3) The laboratory shall document corrective action taken to address the nonconformance under sub. (1) and any other changes resulting from corrective action investigations. Changes implemented to address failures of quality control samples shall be those that resolve or address the failure. Changes shall be implemented to minimize the number of affected results reported by a laboratory.

(4) The laboratory shall monitor the effectiveness of implemented corrective action changes and take additional corrective action when initial or subsequent corrective action fails to resolve the nonconformance.

Note: The analyst may not always be able to identify the cause of isolated nonconformance incidents.

(5) Root cause analysis shall be performed when there is recurrence.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.39 Records and documents. (1) RECORDS AND DOCUMENTS RETENTION AND CONTROL. (a) The laboratory shall establish procedures to control and manage all records and documents that form part of its quality system and that are required to demonstrate compliance with this chapter.

(b) The procedures shall ensure that documents required to perform analyses and to ensure the quality of generated data are available to laboratory personnel, and that records and documents are reviewed periodically for continuing suitability and, when necessary, revised to facilitate compliance with the requirements of this chapter.

(c) The laboratory shall retain all records and documents, which are part of its quality system, and that are required to demonstrate compliance with this chapter, for a minimum of

three years after the generation of the last entry in an associated record or document. The laboratory shall retain records and documents for a longer minimum period if the records and documents are necessary to reconstruct analytical results generated during a three-year period.

(d) The department may require, in writing, that records be retained for a longer period than that specified in par. (c) if the department has initiated legal action involving test results or the accreditation status of the laboratory.

(e) The laboratory shall identify to the department a responsible party for retaining documents and records for the required period in the event the laboratory changes ownership or ceases to be accredited.

(f) Records and documents shall be handled and stored in a manner that ensures permanence and security for the required retention period and that facilitates retrieval to demonstrate compliance with this chapter.

(g) All records shall allow for reconstruction of reported results from raw data. Records and documents shall be legible, and entries shall be safeguarded against obliteration, erasures, overwriting, and corruption and are subject to all the following requirements:

Note: The determination of legibility includes concerns regarding the quality and permanence of records and the ability to decipher numbers and letters. For example, thermal paper ages and eventually becomes unreadable, so thermal paper printouts should ultimately be scanned or copied to ensure permanence.

1. Handwritten records shall be recorded in ink.
2. Records and documents that are stored only on electronic media shall be supported by the hardware and software necessary for retrieval and reproduction into hard copy.
3. Corrections or other alterations made to entries in records or documents may not obscure the original entry.
4. The laboratory shall have procedures to prevent unauthorized access or amendments to records and documents.

(2) ADMINISTRATIVE RECORDS. A laboratory shall maintain all the following administrative records:

(a) Certificates of accreditation issued by the department unless the department has requested a laboratory to return the certificates to the department.

(b) Certificates issued to the laboratory by entities with which the department has entered into a reciprocal agreement under s. NR 149.08, if a laboratory is accredited for this chapter under any existing agreement.

(c) Records of personnel qualifications, experience, and training when personnel are required to possess or maintain specific credentials by s. NR 149.36 (2).

(d) Copies of, or access to, other regulations, standards, and documents necessary for the laboratory to operate or to maintain compliance with this chapter.

(3) REAGENT AND STANDARD RECORDS AND REFERENCE MATERIALS. The laboratory shall document the identity, source, and purity of standards and reagents used in the methods performed. The laboratory shall retain records of reference materials and certificates of analysis when the records are provided by the supplier and are necessary to establish the identity, source, or purity of standards and reagents.

(a) Reagent containers shall be labeled with an expiration date, chemical name, and concentration. Except for instrument vials, standard containers shall be labeled with an expiration date, chemical name, and concentration.

(b) The laboratory shall document the lot number, manufacturer, chemical name, concentration, and the date of expiration for standards and reagents purchased from a manufacturer. These records shall be separate from the container labels.

Note: An expiration date is not required when one is not provided by the supplier.

(c) The laboratory shall document the preparation details of all prepared standards and reagents. These records shall link the prepared standards and reagents to the respective originating stocks or neat compounds and shall indicate the date of preparation, date of expiration, and the identity of the preparer.

(d) The laboratory may not use any standards and reagents beyond the expiration dates unless the laboratory is using the standard and reagents for qualitative determinations.

(e) Certificates for all reference materials shall be maintained.

(4) ANALYTICAL AND TECHNICAL RECORDS. The format of the analytical and technical records of a laboratory shall facilitate access to the information in this subsection and may be contained in bench sheets, log books, notebooks, journals, manuals, standard operating procedures under s. NR 149.40, and forms, in hard copy or electronic media.

(5) SAMPLE COLLECTION RECORDS. The laboratory shall retain records supplied by the collector to allow the laboratory to evaluate collection information against the laboratory's sample acceptance policy.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.40 Standard operating procedures. **(1)** A laboratory shall maintain written standard operating procedures that document or reference activities needed to maintain its quality systems and that enable performing or reproducing an analysis in its entirety as performed at the laboratory. Each laboratory shall develop, maintain, and keep current its standard operating procedures for both sample preparation and analysis.

Note: Sample preparation includes digestions, distillations, extractions, concentrations, dilutions, and clean-up performed on samples prior to the determinative analytical step.

(2) Standard operating procedures may be documents written by laboratory personnel or may consist entirely of copies of published documents, manuals, or procedures if the laboratory follows the chosen source exactly.

(3) Standard operating procedures may consist, in part, of copies of published documents, manuals, or procedures if all the following conditions are met:

(a) Modifications to the published source are described in writing in additional documents.

(b) Clarifications, changes, or choices are completely described in additional documents, when published sources offer multiple options, ambiguous directives, or insufficient detail to perform or reproduce an analysis.

(4) Standard operating procedures shall indicate the dates of issue or revision.

(5) When the standard operating procedure is written by the laboratory, each standard operating procedure shall include, address, or refer to all the following elements, if applicable:

(a) Identification of the referenced method.

(b) For multi-analyte methods, a list of analytes.

(c) Potential interferences and how the interferences are treated.

(d) Equipment and analytical instruments.

(e) Consumable supplies, reagents, and standards.

(f) Sample preservation, storage, and hold time.

(g) Quality control samples and frequency of the analysis.

(h) Calibration and standardization.

(i) Procedure for analysis.

(j) Data assessment and acceptance criteria for quality control measures.

(k) Corrective actions and contingencies for handling out of control or unacceptable data.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.41 Method selection. (1) The laboratory shall use methods for environmental testing required by covered programs under this chapter and that are suitable for the matrix, type of analyte, expected level of analyte, regulatory limit, and potential interferences in the samples to be tested.

Note: Sources, including the following as updated, likely contain methods that are acceptable for testing under this chapter: The EPA, the department, Standard Methods for the Examination of Water and Wastewater, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods – SW-846, American Society for Testing and Materials, and the U.S. Geological Survey Agency.

(2) When methods are not specified by covered programs under this chapter or specified in permits issued by the department, the laboratory shall consult with the department to select a method that meets the requirements in sub. (1).

(3) When using methods associated with the methods compendium document, “Test Methods for Evaluating Solid Waste,” the laboratory shall comply with the minimum requirements of the methods as written and state which options are being implemented when options exist.

Note: The documents above can be found on the Wisconsin department of natural resources laboratory accreditation program website and are available for inspection at the offices of the department and the legislative reference bureau.

(4) The department will assess the scientific validity of method modifications to determine if the modification is within the scope of a method.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.42 Alternative methods. (1) The department may allow the use of alternative methods from those required by covered programs, including the safe drinking water program, if a laboratory requests approval and if the EPA has granted approval for the alternative methods.

(2) On a case-by-case basis, the department may allow the use of methods other than those required by covered programs for any of the following situations:

(a) After consultation with the department, the manager of a covered program determines that the allowance does not result in a detrimental effect on the quality and defensibility of the results to be generated.

(b) The request is for approval of a method that employs a new or emerging technology and there is documentation that substantiates the validity of the new or emerging technology for the intended purpose.

(3) Requests to use an alternative method shall include the reason for seeking the approval, a description of the principles of any new or emerging technology involved, and the potential scope of application of the method. The department may establish criteria for validating the method for the specific application and scope requested. If the laboratory’s method validation results meet the established validation criteria, the department shall allow the use of the method for the specific application and scope requested.

(4) The department shall approve or deny the request for consideration of approval for use within 90 days from the receipt of the request. The department shall consider in its decision whether the covered programs that would be the recipients of the data generated have a demonstrated need for allowing the alternative method.

(5) The department may charge a fee under s. 299.11 (5) (d), Stats., if it is necessary to verify the results of any validation data submitted by a laboratory requesting use of an alternative method.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.43 Laboratory facilities. (1) (ag) The laboratory shall ensure that the environmental conditions of its facility do not adversely affect the required quality of any measurement.

(ar) Laboratory facilities shall ensure effective separation between neighboring areas in which incompatible analytical activities take place. The laboratory shall take measures to prevent cross-contamination.

(b) Access to and use of areas affecting the quality of environmental tests shall be controlled to an extent commensurate with the type of analysis and samples analyzed by a laboratory.

(2) The laboratory shall monitor, control, and record environmental conditions when the environmental conditions are required by the methods or when the environmental conditions influence the quality of test results.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; renum. (1) (intro.), (a) to (1) (ag), (ar) under s. 13.92 (4) (b) 1., Stats., Register February 2021 No. 782.

NR 149.44 Laboratory equipment. (1) GENERAL PROVISIONS. (a) The laboratory shall furnish the equipment necessary and required for the correct performance of all the environmental tests and associated preparations and activities it performs.

(b) The laboratory shall use equipment and software for testing and calibration that achieves the accuracy required to comply with the requirements of the methods or specifications relevant to the environmental testing performed by the laboratory.

(2) LABORATORY SUPPORT EQUIPMENT. (a) The laboratory shall use support equipment only for its intended purpose, and it shall keep that equipment in working order by routine and preventive maintenance.

(b) When support equipment leaves the direct control of the laboratory for maintenance or for any other reason, the laboratory shall ensure that the function and calibration status of that equipment is checked or demonstrated to be in working order before the equipment is returned to service.

(3) CALIBRATION AND VERIFICATION OF SUPPORT EQUIPMENT. (a) The laboratory shall calibrate or verify all support equipment within that equipment’s range of use using available reference materials traceable to NIST. When reference materials traceable to NIST are not commercially available, the laboratory shall use materials of a quality that will ensure the accuracy of the calibrated or verified support equipment for its intended use.

(b) The acceptability criteria for these calibration or verification checks shall be established by the methods, or in the absence, department guidance.

Note: Department guidance can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(c) The laboratory shall establish a procedure for calibrating or verifying the calibration of support equipment which shall include all the following elements:

1. Procedures used for calibrating or verifying the calibration.

2. Procedures for utilization of correction factors when there is a bias.

3. Evaluation criteria used which defensibly documents the continued accuracy of the equipment.

4. Procedures for addressing equipment which fails to meet calibration or verification requirements.

(d) Minimum verification frequencies include all the following:

1. Annually: devices used to measure atmospheric pressure and temperature.

2. Quarterly: mechanical and automatic volumetric dispensing devices, including pipettes.

3. Monthly: balances, with one weight in the expected range of use. Balance weights shall be all the following:

a. Handled and stored in a manner that protects the weights' integrity.

b. Traceable to NIST and of class 2 quality or better. Certified for accuracy every five years by a metrology service outside the laboratory. Alternatively, new weights of class 2 quality or better, traceable to NIST, shall be purchased for use. Weight re-certification shall be performed sooner than every five years if balance checks performed using these weights suggest that a change in the certified weights has occurred.

Note: Weights that are currently NIST traceable may be used to verify other weights.

4. Each day of use: when specific temperatures are required by method, regulation, or covered program, the operating temperature of the equipment used to control temperatures shall be checked and documented.

(e) All the following are exempt from accuracy verification under this section:

1. Glass microliter syringes.
2. Disposable pipettes.
3. Automatic titrator systems.

(4) LABORATORY ANALYTICAL INSTRUMENTS. (a) The laboratory shall use personnel properly trained to operate analytical instruments. Instructions on the use and maintenance of equipment shall be available to instrument operators.

(b) The laboratory shall properly maintain, inspect, and clean all instruments. The laboratory shall establish procedures for the maintenance of analytical instruments to prevent contamination or deterioration that may affect reported results.

(c) The laboratory shall remove from service all analytical instruments that give suspect results or that have been shown to be defective or outside of performance specifications.

(d) When analytical instruments leave the direct control of the laboratory for maintenance or for any other reason, the laboratory shall ensure that the instruments are functional and that a new initial calibration has passed to demonstrate that the instruments are in satisfactory working order before returned to service.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.442 Handling of samples. **(1) SAMPLE ACCEPTANCE POLICY AND SAMPLE HANDLING PROCEDURES.** (a) The laboratory shall have and follow a written policy that clearly outlines the conditions under which samples will be accepted or rejected for analysis or under which associated reported results will be qualified.

Note: Section NR 149.47 (4) provides rejection criteria.

(b) The laboratory shall receive drinking water samples in a secure manner so that the integrity of the sample is maintained.

(c) When samples received do not conform to the descriptions provided by a collector or do not conform to sample acceptance requirements, the laboratory shall consult with the collector or client to determine the proper processing or disposition of the samples.

(d) The laboratory shall place a unique identification code on a sample container as a durable label. The unique identification code shall be used as a link to associate samples with the complete sample history, including treatment and analysis, while in the laboratory's possession.

(2) SAMPLE PRESERVATION AND HOLDING TIME. (a) A laboratory shall follow the sample preservation procedures and holding times required by state and federal regulations.

Note: Sample preservation procedures and holding times are given in 40 CFR Part 136, 40 CFR Part 141, NR 219, SW-846 "Test Methods for Evaluating Solid Waste" and may be specified in the methods.

Note: Links to 40 CFR Part 136, 40 CFR Part 141, NR 219, and SW-846 can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(b) The laboratory shall measure and document the sample temperature at the time of receipt when temperature preservation is required.

(c) The laboratory shall consider any sample requiring preservation at $\leq 6^{\circ}\text{C}$ to be preserved if the sample is received at a temperature greater than its freezing point to 6°C . When samples are received on the same day that they were collected, the samples may not yet have reached the appropriate temperature by the time they arrived at the laboratory. These samples may be considered acceptable, without the need to qualify the data, if all the following apply:

1. Samples were placed on ice at the time of sample collection.

2. Samples were received at the laboratory on ice. "Blue ice" packs may not be considered as received on ice.

(d) When sample temperature measurements are required, the laboratory shall record any of the following:

1. The temperature of an actual sample.
2. The temperature of a temperature blank shipped with the samples.
3. The temperature of the melt water in the shipping container.

(e) The laboratory shall verify the pH of each bottle received for samples requiring chemical preservation to a specific pH requirement under this section. Bottles not received at the proper pH may be adjusted at the laboratory provided that the methods allow preservation upon receipt and the lab retains documentation of its actions.

Note: pH verification is only required from the bottle that is analyzed.

(3) SAMPLE RECEIPT DOCUMENTATION. The laboratory shall document the receipt and condition of all samples in chronological hard copy or electronic records. The records may be maintained in any format that retains all the following information:

(a) The identity of the client or entity submitting samples, or the project associated with the received samples.

(b) The dates of sample collection.

(c) The times of sample collection for samples to be analyzed for tests with holding times expressed in hours.

(d) The unique sample identification code assigned by the laboratory.

(e) Documentation of sample preservation status and other sample conditions on receipt for all sample containers analyzed for those tests for which it is appropriate.

(f) An unequivocal link between the sample identification code assigned by the laboratory and the field collection identification code assigned by the collector.

(g) The requested analyses, unless the laboratory collects and analyzes its own samples and analyses are directed by permit.

(h) The reference to requested test methods when the collector or sample originator specifies the methods.

(i) Any comments resulting from the inspection undertaken to determine whether samples meet the policy in sub. (2).

(4) STORAGE OF SAMPLES. (a) The laboratory shall have procedures and appropriate facilities for avoiding deterioration, contamination, loss, or damage of samples during storage.

(b) The laboratory shall store samples requiring thermal preservations at $\leq 6^{\circ}\text{C}$ at temperatures from greater than the samples' freezing point to 6°C .

(c) The laboratory shall store samples separately from all standards, reagents, food, and other potentially contaminating sources. Samples shall be stored in areas that prevent or minimize cross-contamination.

(d) The laboratory shall store sample extracts, digestates, leachates, or concentrates resulting from any initial preparatory step as specified in this subsection.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.444 Initial instrument calibration requirements. (1) GENERAL PROVISIONS. (a) The laboratory shall calibrate or verify the calibration of all analytical instruments before the instruments are used to provide any quantitative results.

(b) Once a calibration model is selected, a calibration function is established, and an initial calibration is finalized, a laboratory may not change the model or calibration function after samples have been analyzed without performing another initial calibration.

(c) The laboratory shall perform an initial calibration if any of the following apply:

1. After instruments undergo non-routine maintenance.
2. Conditions change the expected behavior of the instrument.
3. When a CCV standard fails and any of the following occur:
 - a. Corrective action taken does not result in a passing CCV standard.
 - b. A second consecutive (immediate) CCV standard is performed under the same conditions and it also fails and the corrective action taken does not result in two consecutive passing CCV standards.

(d) The laboratory shall retain all the raw data necessary to reconstruct or reproduce calibration functions associated with initial calibrations.

(e) For colorimetric technologies, the laboratory may not use a method blank to zero the instrument.

Note: For colorimetric technologies other than those based on inverse chemistries, the instrument is to be zeroed with the matrix of interest which is generally reagent water.

(f) The laboratory may not utilize pre-programmed initial calibrations, provided by the instrument manufacturer, for compliance testing.

(g) The laboratory shall include or reference the details of initial instrument calibration procedures including algorithms, any required equations, and acceptance criteria in the method standard operating procedure.

(h) When required by method, the laboratory shall process each calibration standard in the same manner as samples.

(i) Point-to-point calibrations are not allowed unless otherwise specified in this chapter.

(2) MINIMUM NUMBER OF STANDARDS. To establish calibration, the laboratory shall select the number of nonzero standard concentrations that is appropriate for the calibration model selected and the expected range of concentrations. If a method requires analyzing more than three standards to establish a linear calibration, and the laboratory chooses to narrow the calibration range of the determination to no more than two orders of magnitude, the laboratory may use 3 standards to generate the initial calibration. The minimum number of nonzero standard concentrations selected to establish calibration shall be three except for all the following:

(a) Dissolved oxygen meters, for which the minimum shall be one. Dissolved oxygen meters shall be calibrated against water-saturated air or air-saturated water at a known temperature and

pressure. Alternatively, calibration may be performed using an iodometric method.

(b) Conductivity meters, for which the minimum shall be one. Conductivity meters shall be calibrated by verifying the cell constant or adjusting the meter based on the analysis of a potassium chloride standard solution.

(c) Inductively coupled plasma emission spectrophotometers and inductively coupled plasma mass spectrometers, for which the minimum number shall be one.

(d) pH meters, for which the minimum number shall be two.

(e) Quadratic calibration models, for which the minimum shall be five.

(f) Cubic calibration models, for which the minimum shall be seven.

(3) CONCENTRATION LEVELS OF STANDARDS. The concentration of the standards chosen to establish a calibration function shall be within the same orders of magnitude as the expected concentration of samples.

(4) CALIBRATION MODELS. The laboratory shall select a calibration model that is appropriate for the expected behavior of the analytical instrument to be calibrated. To generate a calibration model, the laboratory shall select a reduction technique or algorithm that is appropriate for the calibration model and the number of nonzero standards used, subject to all the following:

(a) The selected algorithm or reduction technique shall be describable mathematically and shall provide equations, coefficients, or parameters necessary to characterize the calibration function uniquely, unless an analytical instrument is tuned to conform to a universally accepted scientific law or scale.

Note: The response of dissolved oxygen meters is generally adjusted to conform to the concentration of oxygen allowable in a given liquid at a specified temperature and pressure. The response of an ion selective electrode is generally tuned to conform to the Nernst equation. The response of a pH meter is tuned to conform to the universally accepted pH scale. When these instruments are adjusted or tuned according to these principles, characterizing the calibration reduction algorithm mathematically is not necessary.

(b) Non-linear functions may not be used to compensate for instrument saturation, insensitivity, or malfunction.

(c) The laboratory may use weighted algorithms, unless the weighted algorithms are chosen to compensate for deviations from the expected behavior of a detector of an analytical instrument resulting from saturation, insensitivity, or malfunction.

(d) Except for methods that allow average response factors and average calibration factors, the laboratory may not use reiterative reduction techniques or algorithms that force calibration functions through zero.

Note: Reiterative reduction techniques or algorithms that force the calibration function through zero obtain mathematically, by repeated application, a null response for a zero standard that has a nonzero response or adjust calibration parameters to obtain a theoretical null response without analysis of a calibration blank. This paragraph does not prohibit the use of average calibration or response factors or automatic zeroing as part of an initial calibration, when methods, regulations, or covered programs allow those techniques.

(5) EXCLUDING CALIBRATION POINTS. If one or more calibration standards are excluded from the calibration, all the following criteria shall be met:

(a) The rationale for the exclusion is documented.

(b) Any required regulatory limits can still be met.

(c) Except for ICP, ICP/MS, and HRGC/MS, if the highest calibration standard is removed, the linear range shall be limited to the remaining high standard concentration.

(6) EVALUATING ALGORITHM VALIDITY. The laboratory shall establish acceptability criteria for initial calibrations. The type of criteria chosen, and the acceptance range shall be appropriate for the type of analytes to be quantitated, the calibration model selected, and reduction technique or algorithm chosen. Acceptability criteria shall be established using any of the following:

(a) When the x-intercept is used to evaluate the calibration, then the value of the x-intercept of the calibration function for each analyte may not exceed its LOD.

(b) Unless otherwise specified by the method, when RSE is used to evaluate the calibration, the relative standard deviation may not exceed 15% for inorganic analytes or 20% for organic analytes.

(c) Unless otherwise specified by the method, when residuals of each calibration standard are used to evaluate the calibration, the standard recovery for all but the lowest calibration point shall fall within 90% to 110% for inorganic analytes or within 70% to 130% for organic analytes. Recovery for the lowest calibration point shall be within 80% to 120% for inorganic analytes or 50% to 150% for organic analytes.

(d) When average response factors are used to reduce calibration data, the relative standard deviation of the response factors may not exceed 20% unless the method allows a larger percentage.

(e) When linear regression or least squares analysis is used to reduce calibration data, the correlation coefficient (r) of the resultant calibration shall be at least 0.995 for inorganic analytes or 0.99 for organic analytes.

(f) When quadratic (2nd order) or cubic (3rd order) analysis is used to reduce calibration data, the coefficient of determination (r^2) of the resultant calibration shall be at least 0.995 for inorganic analytes or 0.99 for organic analytes.

(7) VERIFYING ACCURACY. Except for calibrations generated using dissolved oxygen meters, pH meters, or conductivity meters, the laboratory shall verify all initial instrument calibrations after the calibrations are generated, but before the calibrations are used to quantitate any samples, with a second source standard, referred to as an ICV standard. ICV standards shall be treated in the same manner as the standards analyzed for the initial calibration. Unless otherwise required by method, regulation, or covered program, the acceptance criteria for the ICV standard shall be all the following:

(a) Obtaining concentrations within 10% of the theoretical concentrations of all reportable inorganic analytes.

(b) Obtaining concentrations within 20% of the theoretical concentrations of all reportable organic analytes.

(8) EVALUATING SENSITIVITY. When methods require an ICB be analyzed after the initial calibration, the ICB shall be treated in the same manner as the initial calibration standards. The concentration of an analyte in an ICB may not exceed its LOD.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (2) (intro.) made under s. 35.17, Stats., Register February 2021 No. 782.

NR 149.446 Continuing instrument calibration requirements. **(1) GENERAL PROVISIONS.** When an initial instrument calibration is not performed on the day of analysis, the continuing validity of the initial calibration shall be verified prior to analyzing any batch quality control or environmental samples by the analysis of one or more CCV standards, subject to all the following:

(a) Except for multi-peak analytes, CCV standards shall contain all analytes to be reported and may be prepared from the same standards used to generate the initial calibration. CCV standards are required for multi-peak analytes when the analytes are detected and reported in the samples.

(b) CCV standards shall be treated the same as the standards used in the initial calibration. When the method requires that the standards be treated the same as samples, the CCV standards

shall be performed with the associated batch so that the CCV standards and samples are all processed together.

(c) Continuing calibration verification is not required for technologies when there are no initial calibrations established.

(d) If an LCS also serves as a CCV standard, the acceptance criteria of the CCV standard shall be used.

(2) FREQUENCY. (a) Continuing calibration verification shall be performed at least once on each analysis day when an initial calibration is not performed and prior to sample analysis and batch quality control analysis.

(b) Continuing calibration verification shall be performed after the consecutive analysis of each group of 20 environmental samples, if 20 or more samples constitute an analytical batch, unless otherwise required by method, regulation, or covered program.

(3) MINIMUM NUMBER OF STANDARDS AND CONCENTRATION LEVELS. (a) For linear and quadratic model calibration functions, the laboratory shall analyze at least a single CCV standard. The concentration of the standard shall be within the range established during the initial calibration.

Note: Linear calibration models include electrometric technologies (pH and ion selective electrode), average response factor, average calibration factor, linear regression, and least squares analysis.

(b) For cubic model calibration functions or third order polynomials, the laboratory shall analyze at least two CCV standards in each instance when a single CCV standard is required by method, regulation, or covered program.

(4) VERIFYING ACCURACY. (a) Unless otherwise required by method, regulation, or covered program, the acceptance criteria for CCV standards shall be within 10% of the theoretical concentrations of all reportable inorganic analytes from an initial calibration.

(b) Unless otherwise required by method, regulation, or covered program, the acceptance criteria for CCV standards shall be within 20% of the theoretical concentrations of all reportable organic analytes from an initial calibration.

(5) ACCURACY CORRECTIVE ACTION. (a) When a CCV standard fails, the laboratory shall do any of the following:

1. Perform corrective action and reanalyze the CCV standard. If the CCV standard does not pass, an initial calibration shall be performed.

2. Perform a second consecutive (immediate) CCV standard under the same conditions. If the second CCV standard also fails, then corrective action shall be performed and two consecutive CCV standards shall pass or an initial calibration shall be performed.

(6) EVALUATING SENSITIVITY. When the method requires that the standards be treated the same as the samples and when the method requires a CCB, the CCB shall be performed with the associated batch so that the CCB and samples are all processed together. The CCB is processed at the same frequency as the CCV standard. The CCB is subject to the same criteria specified in s. NR 149.48 (5) (d).

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.45 Measurement traceability. The laboratory shall maintain all analytical and technical records containing raw and derived data or original observations necessary to allow historical reconstruction of all laboratory activities that contributed to generating reported results. Observations, data, and calculations shall be recorded at the time they are made. At a minimum, the laboratory shall ensure that results of analyses can be linked to sample collection data, preparation records, calibration

records, analytical records, test reports, corrective action, and any chemicals used.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21.

NR 149.47 Reporting results. (1) GENERAL PROVISIONS. (a) The laboratory shall report results of each test performed by the laboratory in accordance with any requirements or instructions specified in the methods or by the department.

(b) The laboratory shall quantitate sample results only from initial instrument calibrations, unless otherwise allowed by method, regulation, or covered program or unless any of the following applies:

1. Samples analyzed by inductively coupled plasma emission spectrophotometers and inductively coupled plasma mass spectrometers having responses at or greater than 90% of the established upper limit of the linear dynamic range of the instruments shall be diluted and reanalyzed.

2. When an analyte does not perform as well as most of the analytes in a multi-analyte initial calibration, analysis may proceed, and results reported for these analytes, provided that the results are appropriately qualified as required in this section.

(c) When samples cannot be diluted and reanalyzed, the laboratory shall report sample results with appropriate qualifiers.

(d) The laboratory shall establish procedures for reporting results for samples analyzed by dual column and dual detector systems. These procedures shall establish all the following prior to analysis:

1. A primary column or primary detector from which results shall be reported.

2. The conditions under which a presumptive identification is confirmed and reported from the secondary column or detector.

(e) When results are greater than the LOQ on dual column or dual detector systems, and the RPD exceeds 40%, then the higher of the two results shall be reported unless the analyst defensibly documents that the higher result is biased due to interference. In this case the laboratory may report the lower result with a qualifier indicating the value of the higher result or report both results.

(f) Excluding microbiological results, MCL exceedances for any regulated analyte associated with ch. NR 809 compliance monitoring shall be reported by the laboratory to the affected water supply facility within 48 hours of completing sample results.

Note: Laboratories performing bacteriological testing for a covered program are certified or approved under ch. ATCP 77 by the department of agriculture, trade, and consumer protection.

(2) FORMAT AND CONTENT. (a) Laboratory test reports shall have formats that facilitate reviewing the content elements specified in this section, unless otherwise provided by pars. (b), (c), and (d). Content elements may be presented in any form, including electronic media.

(b) When tests are performed for internal clients or when a laboratory has a written agreement with a client, the laboratory may issue reports without all the content elements specified in this section. The laboratory shall retain and make available to the department, upon request, records that include the content elements specified in this section.

(c) A laboratory that is operated by a facility whose function is to provide data to monitor the facility's compliance with covered programs shall retain and make available to the department, upon request, records that include the content elements specified in this section. Laboratory reports with all the content elements specified in this section are not required to be issued if any of the following apply:

1. The laboratory is responsible for preparing regulatory reports in a specified format to the department.

2. The laboratory provides information to another individual within the facility for preparation of regulatory reports in a specified format to the department.

(d) Unless otherwise specified by the department, for covered programs that receive data on behalf of a facility, directly from a laboratory, or when provided by pars. (b) and (c), test reports from the laboratory shall include all the following information.

1. The name of the laboratory where the tests were performed.

2. The laboratory's accreditation identification number.

3. The sample identifying information provided by the client or collector.

4. Identification of the methods used for preparation and analysis.

5. The collection date of the samples.

6. Collection, preparation, and analysis times for tests with holding times expressed in hours.

7. The dates of analysis, extraction, or digestion, when a holding time has been established for the preparation step.

8. When non-aqueous sample results are reported, the laboratory shall indicate whether the non-aqueous sample results were reported on a dry weight or wet weight basis.

9. The LOD and LOQ for tests which the department requires reporting to the LOD.

10. Except for HRGC/MS analysis, for sample results requiring adjustments, an indication of whether the LOD and LOQ have been adjusted accordingly.

Note: Sample adjustments are any sample dilutions or sample amounts that were used differently than those used in the initial demonstration of capability and MDL studies.

11. The units of measurement.

12. The date of the test report.

13. Any qualifiers with reported results.

14. The identity of the subcontract laboratory, for each reported result generated by a subcontract laboratory.

(3) AMENDMENTS TO LABORATORY TEST REPORTS. (a) A laboratory may make amendments to a test report already issued by the laboratory in a manner that clearly identifies the reasons for the amendment and that references the original laboratory test report.

(b) Amended reports shall comply with the requirements of this section.

(4) SAMPLE REJECTION OR QUALIFICATION OF RESULTS. The laboratory shall handle results for samples received with insufficient volume to complete the requested analyses, samples received beyond holding time, samples received improperly preserved, samples received in inappropriate containers, or samples received showing evidence that the samples have not been collected according to approved procedures as follows:

(a) Drinking water samples shall be rejected for analysis unless the laboratory has documented instructions from the client to proceed with analyses and all reported results are accompanied by a disclaimer attesting that the results may not be used to determine or evaluate compliance with the safe drinking water act.

(b) Non-drinking water samples shall be rejected for analysis or appropriately qualified.

(5) SAMPLES REQUIRING REANALYSIS OR QUALIFICATION OF RESULTS. Samples shall be re-analyzed, or the affected sample results qualified when any of the following occur:

(a) The concentration of an analyte in the ICB exceeds its LOD.

(b) A CCV standard exceeds limits.

(c) The concentration of an analyte in the CCB or method blank exceeds the criteria specified in s. NR 149.48 (5) (d).

(d) An LCS exceeds limits.

(e) Surrogates or internal standard recoveries exceed limits, unless the failures result from matrix interference, reanalysis is not required but the laboratory shall qualify the results of the affected samples.

(f) When reporting results to the LOD, the concentration of each non-spiked target element in an interference check standard exceeds 10/3 their corresponding LOD for ICP analysis.

Note: The examples for qualifying data listed in this section are common situations. Other situations may exist that could require qualification of data.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (2) (a) made under s. 35.17, Stats., Register February 2021 No. 782.

NR 149.48 Quality control requirements for chemical testing. (1) GENERAL REQUIREMENTS. (a) A laboratory shall establish a quality control program that includes the analysis of appropriate quality control samples and quality control procedures that define their practices. These quality control procedures shall be used to assess all the following:

1. The level of background contamination associated with the preparation and analysis of all samples.
2. The sensitivity of all tests performed.
3. The level of control of an entire analytical system.
4. The bias contributed to sample results by all preparation and analysis steps.
5. The reproducibility of test results.
6. The selectivity of test methods.

(b) A laboratory may not adjust or correct the sample results by the recoveries of associated quality control samples or surrogates unless otherwise allowed by method, regulation, or covered program. A laboratory may not subtract analyte concentrations found in method blanks from sample results unless otherwise allowed by method, regulation, or covered program.

(c) A laboratory shall document deviations from the laboratory's quality system or exceedances of quality control samples. To the extent the department's data systems allow, the deviations shall be communicated with the results.

(d) A laboratory shall establish procedures for identifying and documenting preparation batches that facilitate determining compliance with the frequencies of quality control samples required under this chapter.

(2) LOD. (a) A laboratory shall determine the LOD for all tests performed except for any of the following:

1. Biochemical oxygen demand and carbonaceous biochemical oxygen demand.
2. Tests for which analyzing a fortified sample is impossible or impractical.
3. Titrimetric tests.
4. Gravimetric tests, other than oil and grease as HEM.

(b) A laboratory shall determine the LOD of an analyte annually by 40 CFR Part 136, Appendix B. All sample-processing steps of a method shall be included in the determination of a LOD.

Note: Links to the 40 CFR Part 136, Appendix B can be found on the Wisconsin department of natural resources laboratory accreditation program website.

(c) The LOD shall meet the regulatory limits required by the covered programs.

Note: Exemptions to LOD requirements for specific compounds are provided on the Wisconsin department of natural resources laboratory accreditation program website.

(d) The LOD shall be adjusted when the sample amounts used are different than those used for the LOD determination.

(e) For tests exempted from performing an LOD under par. (a), the laboratory shall establish a reporting limit, or an estimate of a test's sensitivity based on the intended use of the data for a given application.

(f) The LOD shall be determined each time there is a change in a method or instrumentation that affects the sensitivity of an analysis.

(g) For HRGC/MS technology, the estimated detection limit is defined in SW-846 8290A and is equivalent to the LOD.

(3) LOQ. (a) A laboratory shall establish the LOQ for all tests performed except for those exempted from an LOD under sub. (2) (a).

(b) The LOQ shall meet the regulatory limits required by the covered programs.

Note: Exemptions to LOQ requirements for specific compounds are provided on the Wisconsin department of natural resources laboratory accreditation program website.

(c) Except for ICP and ICP/MS single point initial calibrations, the LOQ shall be established as 10/3 the LOD or at the concentration of the lowest standard in the initial calibration. For ICP and ICP/MS, when single point initial calibrations are utilized, the LOQ shall be established as 10/3 the LOD or at the "lower limit of quantitation."

Note: The "lower limit of quantitation" is referenced in SW-846 6010C, 6010D, 6020A, and 6020B.

(d) The LOQ shall be greater than the LOD.

(4) REPORTING LIMITS. (a) Reporting limits are reserved for those analytes exempted under sub. (2) (a) and shall be established based on a test's sensitivity and the intended use of the data.

(b) For biochemical oxygen demand and carbonaceous biochemical oxygen demand, the minimum reporting limit is 2 mg/L which is based on a 300 mL sample volume. When no dilution is equal to 300 mL, the reporting limit shall be adjusted based on the lowest dilution reported.

(c) For total suspended solids, the reporting limit shall be determined using the following formula: Reporting Limit (mg/L) = 1000 / (sample volume filtered in mL).

(5) METHOD BLANK. (a) The laboratory shall process method blanks along with and under the same conditions, including all sample preparation steps, as the associated samples in a preparation batch.

Note: Method blanks are not required for analysis of pH, alkalinity, acidity, conductivity, and solids determinations.

(b) The laboratory shall process method blanks at a frequency of at least one per preparation batch up to 20 environmental samples. When samples are analyzed by methods that do not require a preparation step before analysis, a method blank shall be analyzed at the frequency of one per analytical batch up to 20 environmental samples.

(c) Whenever the concentration of the method blank contains analytes of interest greater than the LOD, the laboratory shall evaluate the nature of the interference and its effect on each sample in a preparation batch.

(d) The acceptance criteria for method blanks are analyte and sample specific and are established based on the highest of any of the following:

1. The LOD.
2. Five percent of the regulatory limit for that analyte.
3. Ten percent of the measured concentration in the sample.

(6) LCS. (a) Unless otherwise exempted by this subsection, the laboratory shall process an LCS at a frequency of at least one sample per preparation batch up to 20 environmental samples, along with and under the same conditions as the associated sam-

ples in a preparation batch. These conditions shall include all sample preparation steps, except for leaching procedure extractions.

Note: TCLP leachates for metals analysis are fortified after the leaching step is completed and before acid preservation.

(b) The laboratory shall fortify the LCS for the biochemical oxygen demand and carbonaceous biochemical oxygen demand tests with a mixture of glucose-glutamic acid as specified in approved methods of analysis. The LCS shall be processed at a frequency of at least one sample per analytical batch for a laboratory that analyzes more than 20 samples per week. A laboratory that analyzes fewer than 20 samples per week shall analyze one LCS per week.

(c) The laboratory is not required to process an LCS for tests for which analyzing a fortified sample is impossible or impractical.

Note: An LCS need not be analyzed for the following tests: pH, solids determinations, chlorophyll a, and color.

(d) The LCS shall be fortified with the analytes specified by method, regulation, or covered program or all reported analytes, except as allowed in par. (e).

(e) For analyses of polychlorinated biphenyls, the laboratory shall fortify an LCS with at least one aroclor per preparation batch. For other tests that determine analytes with responses that encompass more than one chromatographic peak, as in the case of toxaphene and chlordane, the laboratory may fortify an LCS with a single multi-peak analyte per preparation batch. The laboratory shall ensure that all multi-peak analytes detectable by a method are fortified in an LCS at least once every year that any of those analytes are reported at a detectable concentration.

(f) When the method, regulation, or covered program do not specify control limits, the laboratory shall evaluate LCS recoveries and generate in-house control limits, following exclusion of outliers with a statistical technique and using the mean plus or minus 3 times the sample standard deviation. Annually, the laboratory shall review its generated in-house control limits and update those limits whenever the performance characteristics change.

(g) In lieu of using generated in-house control limits for the LCS, the laboratory may opt to use the CCV standard limits.

(7) SELECTIVITY. The laboratory shall establish procedures to confirm the detections of organic analytes determined by technologies that, unlike mass spectrometry or diode array liquid chromatography, do not provide a positive unique identification when a covered program requires it or when the history of a sample source does not suggest the likely presence of the detected analyte.

(a) The laboratory shall develop and document acceptance criteria, which consider retention time shifts, for chromatographic retention time windows.

(b) The laboratory shall document acceptance criteria for mass spectral tuning.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in (2) (e), (3) (a), (4) (a) made under s. 35.17, Stats., Register February 2021 No. 782.

NR 149.50 Technology requirements. The purpose of this section is to establish minimum requirements that can significantly affect data quality but are not always clearly or consistently addressed in all approved methods.

Note: The Department will take the applicability of these requirements into consideration when there are new approved methods or advancements in technology.

(1) OXYGEN DEMAND ASSAYS (BOD OR CBOD). (a) The environmental conditions for the analysis of biochemical oxygen demand and carbonaceous biochemical oxygen demand shall be 17 to 23 °C.

(b) When dissolved oxygen meters are calibrated using a water-saturated air or air-saturated water standard, the laboratory shall verify concentrations in mg/L of those standards by comparing those concentrations to the dissolved oxygen theoretical saturation point. The measured concentration shall be at or near the theoretical saturation point.

(c) The laboratory shall use the theoretical saturation point, based on temperature and barometric pressure, on each day of analysis to assess supersaturation.

Note: When barometric pressure and temperature measurement features are available on the DO meter, they should be taken from the DO meter.

(d) The laboratory shall properly treat supersaturated samples before an initial dissolved oxygen measurement is performed.

(e) When the laboratory uses pipets to deliver sample volumes, the tips shall be manufactured to be wide-bore.

(f) When the laboratory analyzes multiple method blanks and glucose-glutamic acid standards in an analytical batch, each method blank and glucose-glutamic acid standard analyzed shall be assessed individually and associated to the entire analytical batch unless individual method blanks and individual glucose-glutamic acid standards are clearly documented to be traceable to specific groups of 20 samples.

(g) The laboratory shall seed disinfected samples and nitrogenous demand inhibited samples.

(h) The laboratory may not add nitrogenous demand inhibitor to the glucose-glutamic acid standard, to seed material, or method blanks.

(i) The laboratory shall use sample volumes for dilutions that are sufficient to expect 2 mg/L depletion in at least one dilution.

(j) When equipment with multiple dissolved oxygen probes is employed, the laboratory shall calibrate each probe. Sample records shall be traceable to the probe used.

(k) The laboratory shall calibrate dissolved oxygen probes on each day of use.

(L) The laboratory shall use local barometric pressure which has not been adjusted to sea level.

(m) When determining residual chlorine, a minimum detection capability of 0.1 mg/L shall be met.

(2) COLORIMETRIC OR TURBIDIMETRIC. (a) Except for inverse chemistries, the laboratory shall use calibration blanks in the initial calibration of colorimetric or turbidimetric analyses, and those calibration blanks shall be assigned the measured response.

Note: High range chemical oxygen demand and hexavalent chromium are two tests where inverse chemistries are utilized.

(b) When closed vials are digested using block digesters for total phosphorus, the laboratory shall perform the digestion at 150 ± 2 °C for a minimum of 30 minutes.

(c) When the laboratory uses sulfide strips, the sulfide strips shall have a minimum detection capability of 10 mg/L.

(d) The laboratory may not dilute samples after the color reagent has been added to the samples.

(e) The laboratory shall process hexavalent chromium standards the same as samples.

(3) ELECTROMETRIC ASSAYS (I.E. ION-SELECTIVE ELECTRODE). When the laboratory performs electrometric assays, the laboratory shall perform an initial calibration on each day of analysis.

(4) GRAVIMETRIC ASSAYS – RESIDUE (SOLIDS). (a) The laboratory may not use Buchner funnels or Gooch crucibles for determination of total suspended solids or total dissolved solids.

(b) When the laboratory uses pipets to deliver sample volumes for total solids and total suspended solids, the pipet tips shall be manufactured to be wide-bore.

(5) GRAVIMETRIC ASSAYS – OIL & GREASE AS HEXANE EXTRACTABLE MATERIALS (HEM). (a) When using the solid phase extraction technique, the laboratory may not allow polar solvents to contact the sample.

(b) The laboratory shall use activated silica gel for silica gel-treated determinations.

(6) TITRIMETRIC OR POTENTIOMETRIC TITRATION ASSAYS. When standardization is required by method, the laboratory shall standardize all titrants monthly, unless all the following are met:

(a) Unused titrant is never poured back into the original container.

(b) Titrants shall always be protected from light.

(c) LCS recovery control limits shall be set at 90 to 110%, or tighter, and recovery is achieved.

(7) NONDISPERSIVE IR OR MICROCOULOMETRY. (a) For total organic carbon determinations, the laboratory shall perform an inorganic carbon removal check with each analysis batch.

(b) For aqueous samples with results greater than or equal to the LOQ, the laboratory shall perform duplicate injections until the relative percent difference is 10% or less.

(8) ION CHROMATOGRAPHY (IC). The width of the retention time window that the laboratory uses to make identifications shall be based upon measurements of actual retention time variations of standards over the course of a day unless analyst experience provides for another defensible procedure.

(9) FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY (FLAA). (a) The laboratory shall perform at least two consecutive readings for all samples, standards, and quality control samples, and the laboratory shall use the average for calculating results.

(b) When sample concentrations are greater than the LOQ, the laboratory shall use a control limit of 10% or less for the relative percent difference between replicate aspirations.

(c) The laboratory shall include the same acid types and concentrations in calibration standards as those used in samples.

(10) GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETRY (GFAA). (a) The laboratory shall use at least two firings for all samples, standards, and quality control samples.

(b) When sample concentrations are greater than the LOQ, the laboratory shall use a control limit of 10% or less for the relative standard deviation of replicate firings.

(c) When elements are measured at wavelengths lower than 200 nm, the laboratory shall analyze the samples with an instrument equipped with Zeeman background correction or equivalent.

(d) The laboratory shall include the same acid types and concentrations in calibration standards as those used in samples.

(11) COLD VAPOR ATOMIC ABSORPTION SPECTROPHOTOMETRY (CVAA). The laboratory shall ensure that potassium permanganate is present after the two-hour digestion for Hg, or the sample shall be redigested using a smaller sample amount until potassium permanganate remains. Instead, the laboratory could choose to add more potassium permanganate to the affected samples and method blank and digest for an additional two hours.

(12) INDUCTIVELY COUPLED PLASMA EMISSION SPECTROPHOTOMETRY (ICP). (a) The laboratory shall perform a spectral interference identification study before performing any sample analysis using the following single element standards: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na,

Ni, Pb, Sb, Se, SiO₂, Sn, Sr, Ti, Tl, V, and Zn. When other interferences have been identified, the laboratory shall perform appropriate spectral interference identification studies for those interferences. The laboratory shall analyze the interfering elements to identify potential interelement interferences for each mode and wavelength used. This requirement applies to each instrument used for analysis.

(b) The concentration of single element standards used in the spectral interference identification study shall be at or greater than the maximum concentrations encountered in samples.

(c) At the beginning of each analysis day, the laboratory shall verify that interference corrections and background corrections are working properly through the analysis of interference check standards. The interference check standards shall include all the identified interferences at the maximum concentrations encountered in samples.

(d) Interference correction is only valid to the concentration tested in the spectral interference identification study. Samples with interferences present greater than the concentrations tested shall be reanalyzed at a dilution, or if the instrument is capable, the laboratory may analyze a single element standard at the level in the sample to demonstrate that the apparent concentration is less than the LOQ; if it is not less than the LOQ, the interelement correction factors may be updated, and all of the associated data can be reprocessed.

(e) When reporting results to the LOD, the concentration of each non-spiked target element in the interference check standard shall be less than 10/3 their corresponding LOD.

(f) Adjusting background correction shall require re-evaluation of any interference corrections that are affected by the element to which the background correction was made.

(13) INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP/MS). (a) Only those masses listed in methods may be used for identification and quantitation unless the laboratory has supporting data that identifies the potential interfering species for the masses used, and the correction equations needed to resolve the interferences are employed.

(b) All quality control samples shall be performed on the isotope used for identification and quantitation.

(14) GAS CHROMATOGRAPHY (GC). (a) For non-aqueous volatiles analysis, the laboratory shall ensure that the calibration standards contain the same preservative type as the samples, such as methanol, sodium bisulfate, and reagent water.

(b) When the laboratory analyzes multi-peak compounds, such as aroclors, toxaphene, and technical chlordane, the laboratory shall document in its standard operating procedures all the following:

1. For each compound reported, the process used to determine which peaks are used to identify and quantitate the compound.

2. For each compound reported, the process used to determine how the laboratory quantitates the compounds when the compound exhibits weathering, degradation, or positive interferences.

3. For aroclors, the process used to determine how the laboratory quantitates each aroclor when more than one aroclor is present in the sample.

(15) GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC/MS). (a) The laboratory shall meet full scan tune requirements before selective ion monitoring analysis begins.

(b) For non-aqueous samples, the laboratory shall ensure that the calibration standards shall contain the same preservative type

as the samples, such as methanol, sodium bisulfate, and reagent water.

(16) HAZARDOUS WASTE CHARACTERISTICS. (a) The laboratory shall stir samples during pH measurements for toxicity characteristic leaching procedure fluid type determinations.

(b) The laboratory shall perform a flashpoint standard suitable for ignitability determinations for each batch of samples analyzed for flashpoint analysis.

(17) PREPARATORY METHODS. (a) Unless otherwise required by the method, the laboratory shall fortify any quality control sample prior to the addition of the preparation reagents.

(b) The laboratory shall perform microwave preparations with instruments that utilize temperature feedback control.

History: CR 17-046: cr. Register February 2021 No. 782, eff. 6-29-21; correction in numbering in (6) made under s. 13.92 (4) (b) 1., Stats., and correction in (3), (6) (c) made under s. 35.17, Stats., Register February 2021 No. 782.