Chapter NR 149

LABORATORY CERTIFICATION AND REGISTRATION

Note: Chapter NR 149 as it existed on April 30, 2008, was repealed and a new chapter NR 149 was created, Register April 2008 No. 628, effective September 1, 2008.

Subchapter I — General Provisions

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

History: CR 06–005: cr. Register April 2008 No. 628, eff. 9–1–08.

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

(2) Unless otherwise exempted in this section, this chapter applies to laboratories:

(a) Applying for certification and registration.

(b) Holding a certification or a registration.

(c) Submitting data to the department for a covered program.

(d) Generating data that is necessary for the department to determine compliance with a covered program.

Note: Administrative codes and programs requiring analyses to be performed by a certified or registered laboratory are chs. NR 110 – Sewerage Systems, 113 – Servicing Septic Systems, 123 – Well Compensation Program, 131 – Metallic Mineral Prospecting, 132 – Metallic Mineral Mining, 140 – Groundwater Quality, 145 – Private Wells, 150 – Environmental Analysis and Review Procedures, 157 – Management of PCBs, 158 – Hazardous Substance Discharge Notification, 182 – Metallic Mining Waste, 206 – Land Disposal of Municipal and Domestic Wastewaters, 210 – Sewage Treatment Works, 211 – General Pretreatment Requirements, 212 – Wasteload Allocated Effluent Limits, 214 – Land Treatment of Industrial Liquid Wastes, 216 – Stormwater Management, 219 – Analytical Test Methods and Procedures, 347 – Sediment Sampling and Analysis, 507 – Environmental Monitoring for Landfills, 528 – Management of Accumulated Sediment from Storm Water Management Structures, 661 – Hazardous Waste Identification and Listing, 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 for Investigation and Remediation of Environmental Contamination, 712 – Environmental Response Actions, 716 –Site Investigations, 809 – Safe Drinking Water, 811 – Design of Community Water Supplies, 845 – County Administration of NR 812 (Private Wells), and DCF 251 – Group Day Care Centers for Children.

(3) The requirements for the certification of laboratories performing analyses for the safe drinking water program covered by ch. NR 809 are specified in s. NR 149.19.

Note: Laboratories performing analyses for the safe drinking water program covered by ch. NR 809 must be certified even if they do not perform or intend to perform tests commercially for hire. Registration is not available for these analyses.

(4) The requirements for the certification and registration of laboratories performing whole effluent toxicity testing are specified in ss. NR 149.20 and 149.49.

(5) This chapter applies to laboratories analyzing industrial pre–treatment samples when the department is the control authority of a pre–treatment ordinance, or when another control authority requires it.

(6) Laboratories required to perform bacteriological testing for a covered program shall be certified or approved under ch. ATCP 77 by the department of agriculture, trade, and consumer protection.

(7) Laboratories required to perform radiological testing for a covered program shall be certified or approved by EPA.

(8) This chapter establishes requirements that shall be followed, at a minimum, by all laboratories.

(a) Laboratories are also responsible for following any requirements pertaining to analyses and analytical operations contained in mandated test methods or regulations when those requirements are more stringent than the ones specified in this chapter, unless this chapter grants explicit, alternative allowances.

(b) When it is not apparent whether the minimum requirements of this chapter or those specified in mandated test methods or regulations are more stringent, laboratories shall follow the requirements in mandated test methods or regulations.

(c) The department shall retain the authority to make a decision on the stringency of a laboratory requirement when the applicability of a requirement is disputed.

Note: The order of precedence for the authority of a requirement is statute, code, and method. The order of applicability of a requirement is generally method, code, and statute, whenever each succeeding source contains more general or less stringent requirements that are not in conflict.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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.

(2) "Accuracy" means the closeness of a measured value to an accepted reference value or standard.

(3) "Analysis day" means the day in which a specific type of analysis is performed.

(4) "Analyte" means the chemical substance, physical property or organism analyzed in a sample.

(5) "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 certification or registration.

(6) "Analytical balance" means a balance that is capable of measuring masses to at least 4 decimal places.

(7) "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 certification or registration.

(8) "Analytical instruments" means any test instrument used to provide analytical results that is not support equipment.

(9) "Analytical run" means an event consisting of the uninterrupted analysis of a set of samples used to establish the frequency of continuing calibration verification.

(10) "Analytical staff" includes, but is not limited to, laboratory directors, supervisory personnel, quality assurance personnel, technicians, chemists, biologists, personnel performing extractions and analysts.

(11) "Authoritative source" means a publication, text or reference included in Appendix III.

(12) "Aqueous" means a certification or registration matrix designating any aqueous sample that is not a drinking water, and samples with no more than 15% settleable solids.

Note: Samples with more than 10% settleable solids may also be classified as solid.

(13) "Batch" means a set of samples prepared or analyzed together under the same process, instrumentation, personnel, and lots of reagents. An analytical batch refers to a set of any number of prepared samples, such as extracts, digestates or concentrates or samples requiring no preparatory steps analyzed together as a group in an uninterrupted sequence, and may consist of samples of various quality system matrices. A preparation batch refers to a batch of samples, excluding quality control samples, of the same quality system matrix which can be processed simultaneously using the same equipment, reagents and staff. Preparation batch processing shall be completed in a 24−hour period from the start of the processing of the first sample to the start of the processing of the last sample. For laboratories that do not analyze more than 7 samples for a given test and quality system matrix per week, a preparation batch may consist of up to 7 samples, excluding quality control samples, processed during the course of no more than a week.

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

(15) "Blank" refers to a type of quality control sample optimally containing no detectable levels of the analyte or analyte group of interest, typically used to zero an analytical instrument and ensure that any reagents used do not contribute to overall measurements.

(a) "Calibration blank" means a sample containing insignificant or undetectable levels of target analytes used to establish the analytical zero of a calibration function.

(b) "Method blank" means a sample of a matrix devoid of or having a consistent concentration or amount of the analytes of interest processed simultaneously with and under the same conditions, preparatory and analyses steps as the associated samples.

(c) "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.

(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 function" means the specific mathematical relationship established to relate calibration standards to instrument response.

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

(19) "Certification" means the specific form of accreditation extended by the department to laboratories that perform analyses for hire in connection with a covered program, or to laboratories that perform drinking water analyses.

(20) "Certification matrix" means a matrix type that is part of the first tier of a field of certification. Certification matrices are drinking water, aqueous and solids.

(21) "Certified laboratory" means a laboratory that has been granted certification by the department directly or through reciprocal recognition under this chapter.

(22) "Chain of custody" means the procedures and records that document the possession and handling of samples from collection through disposal. A chain−of−custody form is used to document, with a signature, date and time, transfer of the sample from collector to transport/delivery service and then to the laboratory staff receiving the samples. "Evidentiary chain−of−custody" refers to more stringent sample transfer documentation in which samples are stored in secure storage areas. In addition, a chronological written record shall be maintained of all individuals who have possession of the sample from its initial acquisition until its final disposition.

(23) "Coefficient of determination" means a quantity that measures the degree of agreement between the points in a calibration curve and the quadratic function derived to connect them.

(24) "Commercially for hire" means offering analyses for payment or non−monetary compensation generally available to any party requesting analytical services.

(25) "Confirm" means to verify the identity of a compound by an alternative procedure, column, detector, wavelength, or by a technique that bases detection on a different scientific principle from the one originally identifying the compound.

(26) "Control" means to possess, directly or indirectly, the power to direct or cause the direction of the management and policies of an entity, whether that power is exercised through one or more intermediary entities, or alone, or in conjunction with, or by an agreement with, any other entity, and whether that power is established through a majority or minority ownership or voting of securities, common directors, officers, stockholders, voting trusts, holding trusts, affiliated companies, or documented agreements between government entities, whether statewide, countywide, citywide or any combination thereof.

(27) "Control authority" means to have direct or delegated responsibility for establishing, implementing or monitoring an industrial waste pre−treatment program.

(28) "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 them.

(29) "Corrective action" means any measure taken to eliminate or prevent the recurrence of the causes of an existing nonconformity, defect or undesirable condition.

(30) "Council" means the certification standards review council created under s. 15.107 (12), Stats.

(31) "Covered program" means a program defined by s. 299.11 (1) (d), Stats., and includes any department program, project, permit, contract or site investigation that requires analytical work to be performed by a certified or registered laboratory.

Note: Consult the note in s. NR 149.02 (2) (d) for a list of department administrative rules of programs requiring certification or registration under this chapter.

(32) "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.

(33) "Department" means the department of natural resources.

(34) "EPA" means the United States environmental protection agency.

(35) "Field of accreditation" means a unit by which the department grants or recognizes either certification or registration to a laboratory. There are 2 types of fields of accreditation, each consisting of 3 tiers: matrix − analytical technology – analyte or analyte group, and matrix – method – analyte or analyte group.

(a) The matrix – method – analyte or analyte group field of accreditation is limited to the drinking water matrix.

(b) The matrix – analytical technology – analyte or analyte group field of accreditation is available for both aqueous and solid matrices and for either certification or registration.

(c) Registration is available only for aqueous and solid matrices.

(36) "Inert matrix" means a quality control matrix either containing insignificant or undetectable levels of the analytes that will be analyzed in an analytical test. Typical inert matrices are distilled water, deionized water, diatomaceous earth, and Ottawa sand.

(37) "Internal standard" means an analyte added to calibration standards, blanks, quality control and analytical samples as a reference for evaluating and controlling the precision and bias of an analytical test. Responses of internal standards are used to adjust the quantities of analytes reported in tests that employ the standards.

(38) "Laboratory" means a facility that performs tests in connection with a program which requires data from a certified or registered laboratory. A facility consisting of a principal laboratory and annexes within 5 miles of the principal laboratory may be considered a single laboratory at the discretion of the department. When the terms laboratory or laboratories are used unmodified in this chapter, the terms include laboratories certified or registered under this chapter and those seeking certification or registration under this chapter..

(39) "Laboratory control sample" or "LCS" means a sample of an inert matrix 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 is capable of making accurate and precise measurements.

Note: In many EPA methods, the term "lab−fortified blank" is substantially equivalent to a laboratory control sample.

(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. For department purposes, the LOD approximates the MDL and is determined per the method cited in sub. (46).

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

(43) "MCL" means maximum contaminant level and is the maximum permissible level of a contaminant in water which is delivered to any user of a public water system.

(44) "Matrix spike" or "MS" means a sample prepared by adding a known quantity of analyte to an aliquot of an environmental sample and subjecting the sample to the entire analytical procedure to determine the ability to recover the known analyte or compound. The background concentrations of the analytes in the sample matrix shall be determined in a separate aliquot and the measured values in the matrix spike corrected for background concentrations.

Note: In many EPA methods, the term "lab−fortified matrix" is substantially equivalent to a matrix spike.

(45) "Matrix spike duplicate" or "MSD" means a replicate matrix spike prepared and processed in the laboratory in the same manner as its corresponding matrix spike, and generally used to determine the precision of the recovery of an analyte.

(46) "Method detection limit" or "MDL" means the minimum concentration of an analyte that can be measured and reported with 99% confidence that the stated concentration is greater than zero, determined from analyses of a set of samples containing the analyte in a given matrix. The method detection limit is generated according to the protocol specified in 40 CFR 136, Appendix B.

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

(48) "Nonconformance" means a documented or verifiable deviation from the requirements of this chapter.

(49) "On−site evaluation" means an assessment conducted by the department at a laboratory seeking or maintaining certifications or registrations to determine actual or potential compliance with the requirements of this chapter.

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

(51) "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.

(52) "Precision" means the measure of mutual agreement among individual measurements of a sample, usually under prescribed similar conditions, usually expressed as the standards deviation, variance, or range, in either absolute or relative terms.

(53) "Proficiency testing sample" or "PT sample" means a sample obtained from an approved provider to evaluate the ability of a laboratory to produce an analytical test result meeting the definition of acceptable performance outlined in s. NR 149.27. The concentration of the analyte in the sample is unknown to the laboratory at the time of analysis. PT samples are used to evaluate whether the laboratory can produce analytical results within specified acceptance limits.

Note: Proficiency testing samples are also known as performance evaluation samples or reference samples.

(54) "Qualify" means placing a written statement accompanying or referencing test results identifying anomalies or deviations from this chapter encountered in generating the results**.**

(55) "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.

(56) "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.

(57) "Quality control standard" or "QCS" means a solution or sample containing method analyte of known concentration, accompanied by specified analytical acceptance limits, and obtained from a source external to the laboratory and different from the source of calibration standards. These samples are distinguished from proficiency test samples in that the acceptance limits are provided with the sample, rather than after analysis. Quality control standards are used to check either laboratory or instrument performance.

(58) "Quality control limit" means the acceptance criteria used to evaluate for quality control samples. Quality control limits may be those published by the department, referenced in an approved method or calculated by a laboratory.

(59) "Quality system matrix" means a type of sample classification used for establishing quality control acceptance criteria. Quality system matrices include, but are not limited to, drinking water, wastewater influent, wastewater effluent, groundwater, leaching procedure extracts, soils, oils, chemical wastes and biosolids.

(60) "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.

(61) "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, but are not limited to, absorbance, emission counts, area counts, peak heights, abundance and millivolts. Raw data may be stored in hard copy or electronically.

(62) "Reagent grade water" means water which has been treated to remove any impurities that may affect the quality of an analysis.

(63) "Received on ice" means a designation to indicate that sample containers arriving at a laboratory have been received surrounded by an ice slurry, crushed, cubed or chipped ice.

(64) "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.

(65) "Reference standard" means a standard, generally of the highest metrological quality available, from which measurements made at a laboratory are derived.

(66) "Registration" means the specific form of accreditation extended by the department to a laboratory that submits data in connection with a covered program, that does not perform analyses for hire, and that does not perform drinking water analyses.

(67) "Registration matrix" means a matrix type that is part of the first tier of a field of registration. Registration matrices are aqueous and solids.

(68) "Registered laboratory" means a laboratory that has been granted registration by the department directly or through reciprocal recognition under this chapter. A registered laboratory may be a captive industrial laboratory that performs tests solely on its own behalf or that of a subsidiary under common ownership or control, a municipal laboratory owned by a single municipality, or a municipal laboratory owned by more than one municipality that only performs tests for the owning municipalities.

(69) "Relocation" means a move by a laboratory resulting in a change in the laboratory's facility identification number.

(70) "Replicate" means 2 or more substantially equal aliquots analyzed independently for the same parameter.

(71) "Reporting limit" means a concentration or amount of analyte required by the department or client above which numerical results must be reported. Reporting limits may be limits of detection, limits of quantitation, practical quantitation limits or other concentrations, and may be specific to a project or investigation.

(72) "Revocation" means cancellation of a laboratory's certification or registration.

(73) "Results" means the quantitative or qualitative output of an analysis, including, but not limited to, measurements, determinations and information obtained or derived from tests.

(74) "Sample standard deviation" means the standard deviation calculated for a set of samples belonging to a larger population. The sample standard deviation formula contains the quantity "n − 1" in the denominator inside the radical, where n equals the number of samples.

(75) "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.

(76) "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 above a stated quantity.

(77) "Signature" means the name of a person written by that person, or a distinctive mark or characteristic indicating the identity of that person. Signatures can be provided in hard copy or electronically.

(78) "Solid" means a certification or registration matrix designating samples such as soils, sediments, sludges, organic liquids, oils or aqueous products and by−products of industrial processes, and aqueous samples with more than 10% settleable solids.

Note: Samples containing more than 10% but less than 15% settleable solids may also be classified as aqueous.

(79) "Subcontract" means the act of sending a sample or a portion of a sample by a certified laboratory to another certified laboratory.

(80) "Support equipment" means devices that may not be analytical instruments, but that are necessary to support laboratory tests and operations. These devices include, but are not limited to, 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.

(81) "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.

(82) "Suspension" means a temporary cancellation of a laboratory's certification which may not require an on−site evaluation for reinstatement.

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

(84) "Traceability of measurement" means the ability of relating a result or measurement to appropriate state, national or international standards through an unbroken chain of documented comparisons.

(85) "Unfamiliar sample" means a sample for which a laboratory has either no information or questionable information from previous characterizations of samples from the same source. The term unfamiliar also describes a sample for which there is no information about the process generating it.

(86) "Ultra−low level metals" means concentrations of metals at sub−microgram per liter or sub−microgram per kilogram concentrations and those required to be determined in clean room environments.

(87) "Waste characteristic extractions" means extractions, such as the toxicity characteristic leaching procedure, performed on any solid or waste to establish whether it exhibits a defined regulatory characteristic.

(88) "Waste characterization assays" means determinative tests, such as Pensky−Martens closed cup ignitability, corrosivity of liquids and polychlorinated biphenyls screening of organic liquids, performed on any solid or waste to evaluate whether it exhibits a defined regulatory characteristic.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.04 Disclaimers. A laboratory may not claim or imply that data it generates has department approval solely on the basis of the laboratory's certification and registration status.

Note: Certification or registration of a laboratory is not an endorsement by the department of the quality or validity of the data generated by a laboratory. Certification or registration does not guarantee the usability of data generated by a laboratory for an intended purpose. The covered programs under this chapter are the ultimate users of laboratory results and determine whether they accept or reject analytical data from any certified or registered laboratory.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

Subchapter II — Program Administration

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

(2) The department may not accept data required to be generated or submitted by a certified or registered laboratory from a laboratory that is not certified or registered under this chapter except as provided in s. NR 149.11.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.06 Certificates. **(1)** The department shall issue certificates to certified and registered laboratories indicating or making reference to the specific fields of certification or registration for which laboratories have been granted certification or registration. The department shall issue certificates annually and whenever the fields for which a laboratory is certified or registered change, and when a laboratory relocates or changes its name.

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

(a) The department may not issue certificates to an operating contractor of a laboratory 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) Laboratories may not alter or modify certificates issued by the department. Laboratories that alter or modify a certificate, or that misrepresent the fields of certification or registration contained or referenced in a certificate, may be subject to revocation of their entire certifications or registrations.

(5) Certificates shall be displayed conspicuously at the facilities of the laboratories to which they have been issued.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.07 Transfer of certification and registration. **(1)** Laboratory certifications and registrations are not transferable to other entities unless the department expressly approves the transfer.

(2) Laboratories shall notify the department of any change of ownership as soon as practicable, but no later than 30 days after the change has occurred. As part of the notification, the laboratory shall provide the department the number of analytical staff working or expected to be working at the facility 30 days before and after the ownership change.

(3) The department shall inform the laboratory within 30 days after the receipt of the notification or the actual transfer of ownership, whichever happens later, whether the laboratory is eligible for having existing certifications or registrations transferred by application, or whether an initial application is required to be submitted by the new laboratory owner.

(a) The laboratory shall submit the type of application the department has determined is appropriate within 30 days after the date of the determination notification.

(b) All certifications and registrations granted to the laboratory changing ownership shall expire 30 days after the department notifies the laboratory of the type of application required to be submitted.

Note: Requirements for initial and transfer applications are contained in s. NR 149.14.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.08 Recognition of other certifications, registrations, accreditations, licenses or approvals. **(1)** AGRICULTURE, TRADE, AND CONSUMER PROTECTION AGREEMENT. The department shall recognize the certification, registration, 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) EPA AGREEMENT. The department shall recognize the certification, registration, licensure or approval by EPA for radiological testing performed by a laboratory submitting or generating data for a covered program.

(3) LABORATORIES CERTIFIED, REGISTERED, ACCREDITED, LICENSED OR APPROVED BY OTHER GOVERNMENTS. (a) The department shall negotiate with and attempt to enter into agreements with federal agencies and agencies of other states to reciprocally recognize laboratories under this chapter.

(b) The department may recognize the certification, registration, 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 recognize the certification, registration, accreditation, licensure or approval of a laboratory by another state or an agency of the federal government, unless that state or federal agency recognizes laboratories under this chapter.

(4) PRIVATE ORGANIZATION AGREEMENTS. (a) The department may negotiate with and attempt to enter into agreements with private not for profit organizations to recognize laboratories under this chapter.

(b) The department may recognize the certification, registration, 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.09 Certification standards review council. **(1)** The certification standards review council shall advise the department on the standards used to certify, register, suspend and revoke laboratories.

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

(3) The department shall prepare annually for review by the certification standards review council:

(a) A summary of laboratory evaluations performed. The certification standards review council shall advise the department on the frequency and scope of evaluations necessary to determine compliance of laboratories with this chapter.

(b) A list of required proficiency testing samples and available sample providers. The department shall seek the advice of the certification standards review council before requiring the analysis of additional proficiency testing samples and approving sample providers.

(c) A summary of fees scheduled to be assessed to laboratories. The department shall seek the advice of the certification standards review council before implementing changes in the fees assessed to laboratories.

(d) A summary of variances issued. The department shall seek the advice of the certification standards review council in granting variances.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.10 Enforcement. (1) ADMINISTRATIVE PROCEDURES. A laboratory's certification is valid until it expires, is suspended, or revoked. A laboratory's registration is valid until it expires or is revoked. If, after opportunity for a contested case hearing, the department finds that a certified or registered laboratory materially and consistently failed to comply with the provisions of this chapter, the department may suspend or revoke a laboratory's certification or revoke a laboratory's registration by whole or in part by matrix, analytical technology, or analyte or analyte group. Contested case hearings for out−of−state laboratories regulated by 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 quality program requirements as specified in subch. VII.

2. Reporting data to the department after a laboratory is deemed temporarily incapable of performing analysis in any matrix, analytical technology, or 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 quality program requirements as specified in subch. VII.

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 approved 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 2 consecutive proficiency testing samples for any method and analyte or analyte group combination for laboratories holding certification in the drinking water matrix.

8. Demonstrated incompetence manifested by the chronic inability to meet the requirements of this chapter.

9. 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) 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

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

(c) Material and consistent failure to comply with the quality program requirements as specified in subch. VII.

(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) Demonstrated incompetence manifested by the chronic inability to meet the requirements of this chapter.

(4) PROCEDURE FOR SUSPENSION OR REVOCATION OF CERTIFICATION OR REVOCATION OF REGISTRATION. (a) An order suspending or revoking certification or revoking registration 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 certification, the order may include a timetable for correcting the deficiencies that led to the suspension. For orders revoking certification or registration, the department may set a time period for the revocation.

(b) An order suspending or revoking a certification or revoking a registration shall take effect on the thirtieth day after the order is mailed, unless the owner of a certified or registered 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, which 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 10 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 REVOCATION. (a) A laboratory which has had its certification suspended may reapply for certification or registration if all of 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 which has had its certification or registration revoked may reapply for certification or registration 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 period for which the revocation is in effect has expired.

(c) Laboratories reapplying for certification or registration 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 certification or registration for which analysis results are submitted to the department for compliance monitoring or for analyses which require certification or registration under ch. NR 662, 664 or 665, may be referred by the department to the attorney general's office for enforcement.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.11 Discretionary acceptance. (1) The department may accept, on a case−by−case basis, the results of tests originating in a laboratory not certified or registered for fields of certification or registration required by a covered program, if the results meet all other requirements of this chapter.

(2) The department may not accept the results of tests originating in a laboratory not certified or registered for the corresponding fields of certification or registration if the results do not meet all other requirements of this chapter.

(3) The department may not accept the results of tests originating in a laboratory not certified for the corresponding fields of certification for any tests associated with monitoring required under ch. NR 809.

(4) 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 are essential to or have no effect on the department's objectives. Before granting variances, the department shall take into account 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's director of the bureau of integrated science services as far in advance as feasible. Each variance request shall contain:

(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 determination of justification.

(d) A statement as to whether the same or a similar variance has been requested previously, and if so, the circumstances of the previous request.

Note: Requests for variance should be addressed to:
Bureau Director, Science Services
Wisconsin Department of Natural Resources
101 So. Webster Street
PO Box 7921
Madison, WI 53707−7921.

(3) APPROVAL OF VARIANCE. The department shall send a letter approving or denying the requested variance to the applicant within 60 days of receiving all the information referenced in sub. (2). If the request is denied, the letter shall state the reasons for the denial. A copy of all letters approving or denying variances shall be retained in the department's files.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 is offered as either certification or registration. Fields of certification and registration consist of 3 tiers describing the analytical capability of laboratories. Specific fields of certification and registration shall be structured as in Table 1 of this subchapter.

(2) TIER 1−MATRIX. The first tier of certification or registration designates the matrices a laboratory may analyze and shall consist of aqueous, solids, and drinking water matrices.

(3) TIER 2−TECHNOLOGY OR METHOD. The second tier of certification or registration shall be analytical technology for aqueous and solid matrices or method for drinking water matrix.

(a) Laboratories analyzing aqueous and solid samples may be certified or registered for the analytical technologies contained in table 2 of this subchapter.

1. The department shall include any associated sample preparation techniques, such as digestions, distillations, extractions, cleanups, concentration, and dilution as part of the certification or registration for a given field of accreditation.

2. Laboratories may employ multiple approved methods of analysis for a given analytical technology under the same field of accreditation.

Table 1
Fields of Accreditation Tiers

ACCREDITATION TYPE OFFERED

	Certification or Registration	**Certification or Registration**	**Certification only**
Tier 1− Matrix	Aqueous Matrix	Solid matrix	Drinking water matrix
Tier 2− Analytical technology or method	Analytical technology Ex. BOD assay Colorimetric Cold Vapor AA GC/MS	Analytical technology Ex. ICP GC Waste Char. Extn Waste Char. Extn	Method Ex. EPA 200.9 SM 4500 NO3− D EPA 300.0 EPA 524.2
Tier 3− Analyte or Analyte Group	Analyte or Analyte Group Ex. BOD Total Phosphorus Mercury Volatile Organics	Analyte or Analyte Group Ex. Iron PCB (Aroclors) TCLP Ignitability	Analyte or Analyte Group Ex. Arsenic Nitrate Fluoride VOCs

Table 2
Analytical Technologies for Aqueous and Solid Matrices

#	Analytical Technology
1.	Oxygen Demand assays (BOD or cBOD)
2.	Colorimetric or Nephelometric (turbidimetric)
3.	Combustion or Oxidation
4.	Electrometric Assays (i.e. probe, ion−selective electrode)
5.	Gravimetric Assays – Residue (solids)
6.	Gravimetric Assays – Oil& Grease or Hexane Extractable Materials (HEM)
7.	Ion Chromatography (IC)
8.	Titrimetric or Potentiometric Titration Assays
9.	Cold Vapor Atomic Absorption or Gaseous Hydride Spectrophotometry
10.	Flame Atomic Absorption Spectrophotometry
11.	Graphite Furnace Atomic Absorption Spectrophotometry
12.	Inductively Coupled Plasma Emission Spectrophotometry (ICP)
13.	Inductively Coupled Plasma−Mass Spectrometry (ICP/MS)
14.	Ultra−Low Level Metals Assays
15.	Gas Chromatography (GC)
16	Gas Chromatography−Mass Spectrometry (GC/MS)
17	High Resolution Gas Chromatography−Mass Spectrometry (HRGC/MS)
18	High Performance Liquid Chromatography (HPLC)
19.	Liquid Chromatography−Mass Spectrometry
20.	Waste Characterization Extractions[1]
21.	Waste Characterization Assays[2]
22.	Whole Effluent Toxicity Assays[3]
23.	Other[4]

1. Waste characterization extractions offered for solid matrices (Tier 1) only and include extraction procedure toxicity, synthetic precipitation leaching procedure, toxicity characteristic leaching procedure and shake extraction of solid waste with water. Laboratories shall also maintain certification or registration for any analyte to be determined in the resulting extract from any waste characteristic extraction.

2. Waste characterization assays are offered for solid matrices (Tier 1) only and shall include tests required to determine if a material meets the hazardous definition in s. NR 661.03 and those used to fulfill the requirements of waste analysis plans under ch. NR 664 or 665.

3. Certification or registration for this technology is only available for aqueous matrices (Tier 1).

4. The department may offer certification or registration in other analytical technologies if they are approved by EPA or approved by the department as an emerging technology.

(b) Laboratories analyzing drinking water samples shall be certified to perform methods promulgated or approved by the EPA.

1. Methods available for the certification of laboratories analyzing drinking water are contained in ch. NR 809 and the "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA815−R−05−004, fifth edition, EPA, Office of Groundwater and Drinking Water, January 2005.

2. The department may certify laboratories to analyze drinking water using methods not contained in the sources cited in subd. 1. if EPA has promulgated the methods or has granted approval for their use.

(4) TIER 3−ANALYTE OR ANALYTE GROUP. The third tier of the certification fields shall be analyte or analyte group, when the department determines that offering analyte groups improves the efficiency of administering certifications.

(a) The analytes and analyte groups available for certification and registration are contained in appendices I and II.

(b) The department may offer certification or registration for additional analytes or analyte groups that are not contained in appendices I and II upon request by a covered program or when EPA requires their analysis, after consultation with the certification standards review council.

(c) Analyte groups are organized into classes. Laboratories analyzing aqueous and solid matrices may be certified or registered for analyte groups belonging to the analytical classes contained in Table 3 of this subchapter.

Table 3
Classes of analytes groups for aqueous and solid matrices

Number	Class of analyte group
1.	General Chemistry
2.	Metals
3.	Base, Neutral, and Acid Extractable Semivolatile Compounds, including but not limited to: a. Aldehydes and Ketones b. Benzidines c. Chlorinated Hydrocarbons d. Explosive Residues e. Haloethers f. Nitroaromatics and Cyclic Ketones g. Nitrosamines h. Nonhalogenated Organics i. Phenols j. Phthalate Esters
4.	Pesticides and their metabolites, including, but not limited to: a. Acid Herbicides b. Nitrogen c. N−Methyl Carbamates and Substituted Ureas d. Organochlorine e. Organophosphorus f. Triazines g. Pesticides Not Otherwise Specified
5.	Petroleum Hydrocarbons
6.	Polychlorinated Biphenyls (as Aroclors, and as Congeners)
7.	Polychlorinated Dibenzo−p−Dioxins and Furans
8.	Polynuclear Aromatic Hydrocarbons
9.	Volatile Organic Compounds

(d) 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 Table 4 of this subchapter.

Table 4
Classes of Analyte Groups for the Drinking Water Matrix

Number	Class of Analyte Group
1.	Disinfection Byproducts
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.	SOC – Organochlorine Pesticides
8.	SOC – N/P Pesticides
9.	SOC – Herbicides
10.	SOC – Miscellaneous
11.	Trihalomethanes (THM)
12.	Volatile Organic Compounds (VOC)

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08; correction to (4) (d) made under s. 13.92 (4) (b) 1., Stats., Register April 2008 No. 628.

Subchapter IV — Certification and Registration Process

NR 149.14 Application for certification or registration. (1) GENERAL REQUIREMENTS. (a) The certification and registration process requires laboratories to:

1. Submit applications for seeking, revising or transferring certifications or registrations.

2. Declare the fields of certification or registration being sought, revised or transferred in corresponding applications.

3. Declare the methods of analysis that will be used to analyze analyte and analyte groups in the fields of certification or registration being sought, revised or transferred.

4. Submit a current analytical instrument list.

5. Submit acceptable results for proficiency testing samples when the department requires the analysis of these samples.

6. Submit a statement of intent to perform analyses for regulatory samples originating in Wisconsin for laboratories that are not physically located in the state of Wisconsin. Intent to perform analyses for regulatory samples originating in Wisconsin can be manifested by:

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 for a specific field of certification or registration.

8. Allow the department to perform an on−site evaluation, when the department requires it or determines that an evaluation is necessary to determine potential or actual compliance with this chapter.

9. Submit any necessary fees required by this chapter.

10. Agree to comply with this chapter by signing a statement to that purpose in an application.

(b) Laboratories seeking, revising or transferring certifications or registrations shall declare their intent by completing forms provided by the department.

(c) The department may not accept applications seeking, revising or transferring certifications or registrations from laboratories that:

1. Have been issued a notice of violation for nonconformance with this chapter if the nonconformance has not been corrected.

2. Have been issued an administrative order of suspension or revocation for a violation of this chapter when the violation has not been corrected and the suspension or revocation period specified in an order has not elapsed.

3. Are not in compliance with this chapter at the time they voluntarily relinquish their certifications or registrations, the nonconformance existing prior to relinquishing their certifications or registrations has not been resolved, and at least 6 months have not elapsed since the voluntary action was undertaken.

(d) The department shall void any application from laboratories that have not submitted all the information and materials required in an application within a year of the receipt of the application form.

(e) The department may require on a case−by−case basis the submittal with an application of additional information necessary to determine a laboratory's actual or potential compliance with the provisions of this chapter.

(2) INITIAL APPLICATIONS. (a) Laboratories seeking direct certifications or registrations by the department and that have never been certified or registered under this chapter, that have let all their certifications or registrations lapse or expire for more than a year, or that have voluntarily relinquished all their certifications or registrations shall submit initial applications to become certified or registered.

(b) Laboratories seeking certifications or registrations for additional matrices shall submit initial applications for the desired matrices.

(c) Laboratories seeking reinstatement of their certifications or registrations after a suspension or revocation shall submit initial applications for the desired certifications or registrations.

(d) Laboratories seeking to change their valid registrations into certifications shall submit initial applications to effect the conversion.

(e) Laboratories requesting that their certifications or registrations be transferred to a new owner that are ineligible for a transfer shall submit initial applications if they desire to maintain their certifications or registrations. Transfer of ownership transactions involving the purchase or lease of equipment and where less than 60% of the analytical staff are retained are ineligible for transfer of accreditations.

(3) REVISED APPLICATIONS. (a) Laboratories holding valid certifications or registrations shall submit revised applications to seek certifications or registrations in additional:

1. Technologies for a certified or registered matrix.

2. Analytes or analyte groups within a certified or registered analytical technology.

3. Methods for the drinking water matrix.

(b) Laboratories seeking reinstatement of their certifications or registrations within a year after failing to renew them shall submit revised applications for the desired certifications and registrations.

(c) Laboratories seeking to convert their valid certifications into registrations shall submit revised applications to effect the conversion.

(4) APPLICATIONS FOR TRANSFER OF CERTIFICATIONS OR REGISTRATIONS. (a) When the department determines that the valid certifications or registrations of a laboratory are eligible to be transferred to a new owner, the laboratory shall submit an application for transfer of certifications or registrations. Transfer of ownership transactions which do not involve the purchase or lease of equipment and where at least 60% of the analytical staff are retained are eligible for transfer of accreditations.

(b) When the department determines that the valid certifications or registrations of a laboratory are not eligible to be trans-

ferred to a new owner, the laboratory shall submit an initial application to be eligible to retain its certifications or registrations.

(5) APPLICATIONS FOR CERTIFICATIONS OR REGISTRATIONS THROUGH RECIPROCAL AGREEMENT RECOGNITION. (a) Laboratories holding valid certifications, registrations, accreditations, licenses or approvals from government bodies or private organizations with which the department has established a reciprocal agreement may have their certifications, registrations, accreditations, licenses or approvals considered for recognition by the department by submitting reciprocity applications.

(b) Laboratories applying for recognition by the department under an existing reciprocal agreement shall submit certificates or official documents of their certifications, registrations, accreditations, licenses or approvals with their applications.

(c) Laboratories applying for recognition by the department under an existing reciprocal agreement shall agree to notify the department of any changes, within 30 days of their occurrence, in the laboratories' certification, registration, accreditation, licensure or approval status with the entity with which the department has the agreement.

(d) Laboratories applying for recognition by the department under an existing reciprocal agreement shall submit a copy of the report of the last on−site evaluation performed by the entity with which the department has the agreement.

(6) PROCEDURES FOR REVISING CERTIFICATION OR REGISTRATION AS A RESULT OF THE 2007 AMENDMENTS. (a) Prior to September 1, 2008, the department shall provide and the laboratories shall complete and submit a one−time status update form to facilitate the conversion of the test categories and demonstrate that the requirements of s. NR 149.15 (2) have been met. The department may not assess fees for the conversion to the amended program structure in s. NR 149.13.

(b) The purpose of the status update form is to convert current certifications or registrations under the existing program structure into equivalent certifications or registrations under the revised program structure. The status update form may not be used to add additional analytes or analyte groups to a laboratory's list of certifications or registrations. If the laboratory wishes to become certified or registered in additional test categories, the laboratory shall comply with provisions of s. NR 149.14 (3). The laboratory may apply for the additional test categories on the status update form.

Note: Status update forms will be provided to all participating laboratories and will be made available on the department's website at http://www.dnr.state.wi.us/org/es/science/lc/APPLICATION/AppForms.htm.

(7) ISSUANCE OF CERTIFICATIONS OR REGISTRATIONS. (a) The department shall issue certifications and registrations 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 reciprocal application for certification or registration 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.

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 certification or registration, on a case−by−case basis, for selected fields of certification or registration under application in fields that are unaffected by any deficiencies encountered during the on−site evaluation.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.15 Period, renewal and expiration of certification or registration. (1) CERTIFICATION AND REGISTRATION PERIOD. (a) The certification and registration period shall commence on September 1 and end on August 31 of the following year for all laboratories certified or registered by the department.

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

(2) RENEWAL PROCESS. (a) Prior to September 1 of each year, each directly certified or registered laboratory shall:

1. Pay the required annual renewal fee and any assessed administrative fees.

2. Submit acceptable proficiency testing sample results as required in subch. V.

(b) Prior to September 1 of each year, each laboratory that is certified or registered through a reciprocal agreement shall:

1. Pay the required annual renewal fee and any assessed administrative fees.

2. Submit certificates or official documents of their certifications, registrations, accreditations, licenses or approvals from the entity with which the department has the agreement.

3. Submit a copy of the most recent on−site evaluation report from the entity with which the department has the agreement.

(3) EXPIRATION OF CERTIFICATIONS OR REGISTRATIONS. (a) The department shall void on September 1 of each year the certifications or registrations of laboratories failing to provide the information and fees specified in sub. (2) (a).

(b) The department shall void on September 1 of each year the certifications or registrations of laboratories certified through an existing reciprocal agreement that fail to provide the information and fees specified in sub. (2) (b).

(4) VOLUNTARY WITHDRAWAL OF CERTIFICATIONS OR REGISTRATIONS. Laboratories may voluntarily withdraw certifications or registrations at any time by notifying the department in writing.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.16 Notification of relocation. (1) Laboratories relocating shall notify the department in writing, at least 30 days prior to the relocation, of their change of address and any changes in their contact information.

(2) The department shall issue a revised certificate to a relocating laboratory within 30 days of receiving the notification or the effective date of the relocation, whichever is later.

(3) Laboratories undergoing a change of ownership, needing to add certifications or registrations, modifying their certification or registration status, changing the entity by or through which they obtained certifications or registrations as a result of a relocation shall comply with the requirements of s. NR 149.14.

(4) The department may perform an on−site evaluation of the relocating laboratory at its new location to determine the laboratory's continued ability to comply with the requirements of this chapter.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.17 Laboratory name change. (1) Laboratories that change names without changing ownership shall notify the department in writing within 30 days of the effective date of the name change.

(2) The department shall issue a revised certificate to a laboratory changing its name without changing ownership and not seeking additional certifications or registrations within 30 days of receiving notification from the laboratory.

(3) The department may not charge a fee for processing laboratory name changes or for issuing a revised certificate resulting solely from a name change.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.18 Subcontracting of analyses by certified or registered laboratories. (1) Laboratories needing or desiring to have samples they have received or for which they are

responsible be analyzed by another laboratory shall only have the associated samples analyzed in laboratories that have valid certifications or registrations under this chapter.

(2) Laboratories 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 and the department upon request.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.19 Requirements for certification in the drinking water matrix. This section applies to laboratories analyzing drinking water for compliance with the safe drinking water program and that analyze drinking water samples in support of the compliance monitoring required by ch. NR 809.

(1) GENERAL REQUIREMENTS. (a) The minimum criteria and procedures for certification in the drinking water matrix are specified in Chapters III and IV of the "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA815−R−05−004, fifth edition, EPA, Office of Ground Water and Drinking Water, January 2005, except that:

1. The department may not grant provisional certification to laboratories.

2. The department may not grant interim certification to laboratories.

3. Laboratories shall analyze drinking water replicates or matrix spike duplicates at a frequency of one pair per preparation batch or one per 20 analytical samples in an analytical batch.

(b) Laboratories shall follow any additional criteria and procedures identified in this chapter applying to drinking water analyses.

(2) REQUIREMENTS FOR INORGANIC CONTAMINANTS. To receive certification to conduct analyses of inorganic contaminants, the laboratory shall achieve the method detection limits specified in 40 CFR 141.23 (a) (4) (i) and 40 CFR 141.89 (a) (1) (iii) or 10% of the MCL, for contaminants having an MCL, whichever is greater, for each method of analysis.

(3) REQUIREMENTS FOR VINYL CHLORIDE. To receive certification to conduct analyses of vinyl chloride, the laboratory shall achieve a method detection limit of 0.0003 mg/L for each method of analysis.

(4) REQUIREMENTS FOR OTHER VOLATILE ORGANIC COMPOUNDS. To receive certification to conduct analyses of volatile organic compounds, excluding vinyl chloride, but including trihalomethanes, the laboratory shall achieve method detection limits of 0.0005 mg/L for all regulated compounds for each method of analysis.

(5) REQUIREMENTS FOR SYNTHETIC ORGANIC CONTAMINANTS. To receive certification to conduct analyses of synthetic organic contaminants, the laboratory shall achieve the method detection limits specified in 40 CFR 141.24 (h) (18) or 10% of the MCL, whichever is greater.

(6) EXCLUSIONS FROM REQUIRED CERTIFICATION. Certification is not required to perform any of the following analyses:

(a) Fluoride analysis required under ch. NR 809.

(b) Analysis for free chlorine residual and total chlorine residual required under s. NR 809.563 (2) Table R.

(c) Analysis for pH required under s. NR 809.113 (1) Table A 21.

(d) Analysis for turbidity required under s. NR 809.113 (1) Table A 27.

(7) NOTIFICATION TO AFFECTED WATER SUPPLY FACILITIES. Laboratories certified under this chapter for the drinking water matrix shall notify water supply facilities that an MCL exceedance has occurred no later than 48 hours after completing analyses whenever compliance samples exceed an MCL for any regulated analyte under ch. NR 809.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08; **corrections made in (6) (b), (c), (d) made under s. 13.92 (4) (b) 7., Stats., Register April 2011 No. 664.**

NR 149.20 Requirements for certification or registration in the whole effluent toxicity analyte class. This section applies to laboratories certified or registered in the aqueous matrix that perform whole effluent toxicity testing.

(1) GENERAL REQUIREMENTS. (a) The criteria and procedures for the certification or registration of laboratories performing whole effluent toxicity testing are specified in table A of s. NR 219.04.

Note: Method for analyses for determining the toxicity of effluents are referenced in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual", 2nd edition. This document can be obtained at www.dnr.state.wi.us/org/water/wm/ww/biomon.

(b) Laboratories shall follow the requirements for quality systems specified in ss. NR 149.36 to 149.49.

(2) CHEMICAL TESTING IN SUPPORT OF WHOLE EFFLUENT TOXICITY TESTING. (a) Any laboratory performing tests for alkalinity, ammonia and hardness conducted in support of regulatory samples analyzed for whole effluent toxicity need not be certified or registered for those tests if the laboratory is certified or registered for performing whole effluent toxicity testing.

(b) Laboratories that are not certified or registered for performing whole effluent toxicity testing shall be certified or registered for performing tests for alkalinity, ammonia and hardness when those tests are undertaken in support of regulatory samples analyzed for whole effluent toxicity.

(c) Laboratories need not be certified or registered to perform tests for pH, conductivity, dissolved oxygen and total residual chlorine, when those tests are undertaken in support of regulatory samples analyzed for whole effluent toxicity.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.21 Fees. The department shall set a schedule of fees for laboratories participating in the program that is designed to recover the costs of administering this chapter. 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 program's total fee income shall be designed to generate revenues equal to the department of administration's approved spending authority for this program. Any amendments to the formulas in this subsection shall be reviewed by the laboratory certification standards review 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 laboratory certification standards review council and approved annually by the natural resources board.

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

1. Fee Income $\leq$ ASA − TR.

a. Fee income is the total of all fees, including application fees, renewal fees and late fees, that are collected in a given fiscal year.

b. TR is the total out−of−state travel reimbursement in a given fiscal year.

c. ASA is the approved spending authority for the given fiscal year. The department may substitute a lesser amount than the ASA if the ASA is greater than the estimated costs of the program.

d. Estimates of the fee income and travel reimbursement shall be calculated according to s. NR 149.21 (1) (d).

Note: The department of administration approved spending authority is given in s. 20.7379 (2) (fj), Stats., and may be revised by the department of administration to recover program cost.

2. Total # RV Units = Σ (# Laboratories in Item) (RV of Item).

a. Total # RV Units is the total number of relative value (RV) units available for the fiscal year. The relative value units for each fee item (RV of item) are listed in tables 1, 2 and 3.

b. # Laboratories in item is a count of how many laboratories paid the fee for that item for a given fiscal year.

c. Total # of RV Units is calculated by summing the product of (RV of item) and (# laboratories in each item) for each item.

3. Cost per RV = (ASA − TR)/Total # RV Units. The Cost per RV is the dollar value assigned to one RV unit.

4. Cost of Item = (RV Unit of Item) (Cost per RV).

(d) The fees for the upcoming fiscal year shall be based upon program information from the previous fiscal year and upon the approved spending authority for the upcoming fiscal year. The number of laboratories participating in the program shall be determined no earlier than 6 months prior to the billing for the upcoming fiscal year. The estimated travel reimbursement shall be equal to the travel reimbursement from the preceding fiscal year. The calculated fees may not be adjusted during the current fiscal year once laboratories have been billed.

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

Table 1
Administrative Fees

Item	Relative Value Units
Discretionary Acceptance (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 program for travel costs incurred by the cancellation or postponement of an evaluation, not limited to airfare, hotel and rental car expenses.

[2] Assessed 30 days after payment due date.

(3) APPLICATION FEES. The department shall assess fees for all applications specified in Table 2 of this subchapter. 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 certifications or registrations.

Table 2
Application Fees

Item	Relative Value 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 certifications or registrations under this chapter either directly or through recognition agreements. A laboratory's annual fee shall be the sum of all of the following:

(a) The base fee for certification or registration. The department shall assess a base fee to all laboratories holding certifications or registrations under this chapter. The number of relative value units assigned to each type of base fee is specified in Table 3 of this subchapter

(b) The matrix fee. The department shall assess a fee per matrix type to all certified and registered laboratories. The number of relative value units assigned to each type of matrix fee is specified in Table 3 of this subchapter.

(c) Analytical technology or analytical class fees, considering any maximum specified in this subsection.

1. 'Analytical technology fees.' The department shall assess a fee for each analytical technology per matrix to all certified and registered laboratories in fields involving the aqueous and solid matrices. The assessed fee shall be based on the relative value units specified in Table 3 of this subchapter and subject to any maximum fee specified in this subchapter.

a. The maximum analytical technology fee assessed to any lab for the aqueous matrix shall be 22 relative value units (RVU).

b. The maximum analytical technology fee assessed to any lab for the solid matrix shall be 22 relative value units (RVU).

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 relative value units specified in Table 3 of this subchapter and subject to any maximum fee specified in this subchapter. The maximum analytical class fee assessed to any lab for the drinking water matrix shall be 31 relative value units (RVU).

(d) Any outstanding administrative fees.

Note: Considering base fees, matrix fees, analytical technology fee maximums, and the analytical class fee maximum, this effectively establishes a maximum annual fee "cap" of 100 RVUs for any laboratory.

Table 3
Annual Fees for Certification and Registration

	Item	Relative Value Units
A.	**Administrative Fees**	
	Outstanding administrative fees	per Table 1 of this subchapter
B.	**Base Fees**	
	Base Fee, Certification	10
	Base Fee, Registration	5
C.	**Matrix Fees**	
	Matrix Fee, Aqueous	5
	Matrix Fee, Drinking Water	5
	Matrix Fee, Solids	5
D.	**Analytical Technology Fees for Aqueous and Solid Matrices**	
	Electrometric Assays (ion−selective electrodes)	1
	Gravimetric Assays, Residues (solids)	1
	Gravimetric Assays, Oil and Grease (HEM)	2
	Titrimetric or Potentiometric Titration Assays	1
	Colorimetric or Nephelometric Spectrophotometry	2
	Combustion or Oxidation	2
	Oxygen Demand assays (BOD, cBOD)	3

	Ion Chromatography	3
	Waste Characteristic Extractions (*Solid Matrix only*)	1

Table 3 – Continued
Annual Fees for Certification and Registration

	Item	Relative Value Units
	Waste Characterization Assays (*Solid Matrix only*)	1
	Flame Atomic Absorption Spectrophotometry	2
	Cold Vapor Atomic Absorption or Gaseous Hydride Spectrophotometry	3
	Graphite Furnace Atomic Absorption Spectrophotometry	3
	Ultra−Low Level Metals Assays	3
	Inductively Coupled Plasma Emission Spectrophotometry	4
	Inductively Coupled Plasma−Mass Spectrometry	5
	Gas Chromatography	3
	Gas Chromatography−Mass Spectrometry	4
	High Performance Liquid Chromatography	3
	Liquid Chromatography−Mass Spectrometry	4
	High Resolution Gas Chromatography−Mass Spectrometry	10
	Whole Effluent Toxicity Assays (*Aqueous Matrix only*)	5
	Other	Not to exceed 10[1]
E.	**Analytical Class Fees for Drinking Water Matrix**	
	Disinfection Byproducts	5
	Primary Inorganic Contaminants (Non−Metals)	3
	Primary Inorganic Contaminants (Metals)	6
	Secondary Contaminants (Non−Metals)	2
	Secondary Contaminants (Metals)	3
	SOC – Dioxin	8
	SOC – Organochlorine Pesticides	3
	SOC – N/P Pesticides	3
	SOC – Herbicides	3
	SOC – Miscellaneous	4
	Trihalomethanes (THM)	2
	Volatile Organic Compounds (VOC)	4

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

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

Subchapter V — Proficiency Testing

NR 149.22 Required analyses of proficiency testing samples. (1) REQUIREMENTS. (a) Laboratories shall participate in at least one single−concentration proficiency testing study per certification or registration period for each analyte or analyte group identified by the department as specified in sub. (2).

1. For aqueous and solid matrices, laboratories shall analyze aqueous matrix proficiency testing samples for each combination of technique and analyte or analyte group in a laboratory's fields of certification or registration.

2. For the drinking water matrix, laboratories shall analyze proficiency testing samples for each combination of method and analyte or analyte group in a laboratory's fields of certification.

(b) Single−concentration proficiency testing studies may be those offered at set intervals by proficiency testing sample providers, "rapid response" samples or custom formulations approved by the department.

(2) LISTS OF REQUIRED PROFICIENCY TESTING SAMPLES AND APPROVED PROVIDERS. (a) The department shall publish a list of required proficiency testing samples and approved proficiency testing sample providers annually. The department shall seek the advice of the certification standards review council prior to identifying required proficiency testing samples and approved sample providers.

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

Note: Lists of required testing samples and approved proficiency testing sample providers are available on the department's website at http://www.dnr.state.wi.us/org/es/science/lc/PT/Index.htm.

(3) EXEMPTIONS. (a) Laboratories performing the following analytical techniques for metals analysis in aqueous and solid matrices shall analyze quality control standards 3 times per year at evenly spaced intervals in lieu of analyzing proficiency testing samples:

1. Flame atomic absorption spectrophotometry.

2. Colorimetric, for analytes other than hexavalent chromium.

(b) Laboratories analyzing ultra−low level metals in aqueous and solid matrices shall analyze quality control standards 3 times per year at evenly spaced intervals in lieu of analyzing proficiency testing samples. Quality control standards shall be diluted to fall within the working concentration of the analytical technique.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.23 Approval of proficiency testing sample providers. When evaluating a proficiency testing sample provider for approval, the department shall consider criteria including, but not limited to, the provider's:

(1) Accreditation status by nationally recognized accreditation programs.

(2) Use of techniques for calculating acceptance limits as specified in s. NR 149.27.

(3) Ability to submit results to the department in a format specified by the department, including electronic media.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.24 Schedule of analysis. (1) APPLICATIONS FOR AQUEOUS AND SOLID MATRICES. Laboratories submitting initial or revised applications for certification or registration in aqueous

and solid matrices shall analyze proficiency testing samples from an approved proficiency testing sample provider and submit acceptable results for each technique and analyte or analyte group for which the department has identified that proficiency testing samples are required.

(a) Acceptable proficiency testing samples shall be analyzed no more than 6 months prior to the date of application.

(b) The department may not grant a certification or registration unless the associated proficiency testing sample results meet the criteria specified in s. NR 149.27.

(2) APPLICATIONS FOR DRINKING WATER MATRIX. Laboratories submitting initial or revised applications for certification in the drinking water matrix shall analyze proficiency testing samples from an approved proficiency testing sample provider and submit acceptable results for each method and analyte or analyte group.

(a) Acceptable proficiency testing samples shall be analyzed no more than 6 months prior to the date of application.

(b) The department may not grant a certification or registration unless the associated proficiency testing sample results meet the criteria specified in s. NR 149.27.

(3) ANNUAL RENEWAL FOR AQUEOUS AND SOLID MATRICES. Laboratories seeking renewal of certification or registration for aqueous or solid matrices shall analyze at least one proficiency testing sample, prepared in an aqueous matrix, from an approved proficiency testing sample provider and submit acceptable results for each technique and analyte or analyte group for which the department has identified that proficiency testing samples are required. Laboratories with 3 consecutive proficiency testing sample failures in a year for any technique and analyte or analyte group shall submit 2 consecutive acceptable proficiency testing samples from an approved proficiency testing sample provider for that technique and analyte or analyte group.

Note: Proficiency testing samples prepared in a solid matrix are not required at this time to obtain or renew certification or registration for analytes or analyte groups under the solid matrix tier.

(a) For renewal of certifications or registrations, which begin on September 1 of each calendar year, acceptable proficiency testing sample results shall have been reported by an approved PT provider no sooner than January 1 or later than August 15 of the same calendar year.

Note: For example, to renew certification for any parameter effective for the period from September 1, 2009 to August 31, 2010, a laboratory shall have successfully analyzed a PT sample for that parameter reported between January 1 and August 15, 2009.

(b) Reports from proficiency testing sample providers shall be received by the department on or before August 15 of each year.

(c) The department may not renew a certification or registration unless the associated proficiency testing sample results meet the criteria specified in s. NR 149.27.

(4) ANNUAL RENEWAL FOR DRINKING WATER MATRIX. Laboratories seeking renewal of certification for the drinking water matrix shall analyze at least one proficiency testing sample from an approved proficiency testing sample provider and submit acceptable results for each method and analyte or analyte group.

(a) For renewal of certifications or registrations, which begin on September 1 of each calendar year, acceptable proficiency testing sample results shall have been reported by an approved PT provider no sooner than January 1 or later than August 15 of the same calendar year.

(b) Reports from proficiency testing sample providers shall be received by the department on or before August 15 of each year.

(c) The department may not renew a certification or registration unless the associated proficiency testing sample results meet the requirements specified in s. NR 149.27.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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

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

(2) Laboratories may report multiple results of multiple analyses of a single proficiency testing sample when a laboratory maintains certifications or registrations for multiple techniques for any analyte or analyte group in aqueous and solid matrices.

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

(4) Prior to submitting proficiency testing results to a proficiency testing sample provider:

(a) Laboratories may not send a proficiency testing sample, or portion of a proficiency testing sample to another laboratory for analysis.

(b) Laboratories may not knowingly analyze a proficiency testing sample, or a portion of a proficiency testing sample from another laboratory.

(c) Laboratories may not communicate results of a proficiency testing sample with another laboratory.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.26 Submittals. (1) Laboratories shall submit proficiency testing sample results to providers in accordance with the dates specified by the providers.

(2) Proficiency testing reports may be submitted to the department directly from the 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. Reports submitted by the laboratory shall be submitted in their entirety, without modification, to the department.

(3) Results from all proficiency testing reports issued to the department by providers shall be used to determine a laboratory's certification or registration status.

(4) Proficiency testing reports may be amended and reissued by the provider when errors attributable to the proficiency testing sample provider are identified. The department shall accept amended and reissued reports if they are:

(a) Clearly labeled as revised or reissued.

(b) Directly submitted to the department by the provider.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 proficiency testing sample.

(c) The laboratory does not report a result for an analyte present in the proficiency testing sample.

(d) The laboratory fails to submit its results to the proficiency testing sample provider on or before the deadline for the proficiency testing study.

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

(b) For required proficiency testing sample analytes in aqueous and solid matrices for which EPA has not developed acceptance limits, the department may develop acceptance limits based on its experience or information supplied by approved providers.

(c) When an insufficient number of laboratories participate in a study to generate peer−based acceptance limits in a proficiency testing sample with analytes for which EPA has not established

acceptance limits, the department may grade results using fixed acceptance limits.

(d) Proficiency testing 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 (f)(19)(i)(A) and (B) and 40 CFR 141.89 (a)(1)(ii), or developed by EPA.

(e) Where certification or registration in an analyte group is based on passing a representative proficiency testing 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.

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

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.28 Procedure for correcting unacceptable proficiency testing sample results. (1) AQUEOUS AND SOLID MATRICES. If a laboratory does not meet the acceptance limits for a particular analyte or analyte group and the laboratory does not have acceptable results on a previous sample analyzed during the same certification or registration period, the department shall require the laboratory to analyze a second proficiency testing sample for that analyte or analyte group.

(a) If the results of a second proficiency testing 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. After failing 2 consecutive proficiency testing samples, the laboratory shall:

1. Submit 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 proficiency testing sample.

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

(b) The department may not renew the certification or registration of those analytes or analyte groups for which a laboratory has failed 3 consecutive proficiency testing samples for those analytes or analyte groups and has not successfully analyzed 2 subsequent and consecutive proficiency testing samples for those analytes or analyte groups prior to September 1.

(c) When applying to have an analyte or analyte group reinstated after non−renewal for failing 3 consecutive proficiency testing samples, the laboratory shall provide acceptable results on 2 subsequent and consecutive proficiency testing sample studies for that analyte or analyte group.

(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 proficiency testing 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.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 3 years and:

(a) When a laboratory submits an application to become certified or registered in any field of certification or registration, 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 and satisfactory.

(b) When a laboratory changes its location, 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 the change in location.

(c) When 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) When 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.30 Evaluation procedures and appraisal. (1) The department shall perform on−site evaluations of laboratories according to documented procedures that promote consistency in determining a laboratory's potential, actual or continued ability to comply with this chapter.

(2) The department shall provide forms that allow laboratories to voluntarily appraise the evaluation process.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.31 Evaluation reports. (1) The department shall document the deficiencies of an on−site evaluation 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 within 10 days after the conclusion of the 30−day period about the delay. The notice shall include an expected delivery date for the report.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.32 Evaluation corrective action. (1) A laboratory shall take corrective action to address any deficiencies discovered during an on−site evaluation.

(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 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.

(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 180 days of the conclusion of on−site visit that the evaluation process has been completed.

(b) When the department determines that additional action or documentation is needed to evaluate compliance with this chap-

ter, the department shall agree on a date for a second corrective action plan to be submitted in consultation with the laboratory.

1. When the department determines that the second corrective action plan addresses all noted deficiencies satisfactorily, the department shall inform the laboratory in writing that the evaluation process has concluded.

2. When the department determines that the second corrective action plan 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. When a second on−site evaluation is scheduled as a follow−up to a second corrective action plan, 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 them 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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. Laboratories shall conduct their analytical activities under a quality system that incorporates the provisions of this subchapter. At least one individual, however named, 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.36 Laboratory personnel. (1) MANAGEMENT AND ANALYTICAL STAFF. The laboratory shall have personnel with education, training or experience that allows them to comply with the requirements of this chapter.

(2) PERSONNEL INVOLVED IN DRINKING WATER ANALYSES. Additional education and training requirements of management and analytical staff involved in analyzing drinking water are contained in Chapters III and IV of the "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA 815−R−05−004, fifth edition, EPA, Office of Groundwater and Drinking Water, January 2005.

Note: This document is available at: http://www.epa.gov/safewater/labcert/labindex.html.

(3) DEMONSTRATION OF CAPABILITY. (a) When laboratories reference methods that contain protocols for demonstrating initial capability, continuing capability or both, personnel performing analyses using these methods shall perform the protocols, meet any associated evaluation criteria and document results.

(b) When a laboratory references an analytical test method that does not contain protocols for demonstrating initial capability, continuing capability or both, the laboratory shall establish 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 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.37 Quality manual. (1) PURPOSE AND GENERAL PROVISIONS. The laboratory's quality system shall be defined in a quality manual, however named. 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, however conceived, 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.

Note: Although this section does not require a specific format for quality manuals, the format suggested by the following is acceptable to the department: "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA 815−R−05−004, fifth edition, EPA, Office of Groundwater and Drinking Water, January 2005. This document is available at: http://www.epa.gov/safewater/labcert/labindex.html.

(3) CONTENT. The quality manual shall include, address or refer to, at a minimum, the following elements:

(a) Organization and management structure of the laboratory.

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

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

(d) Procedures for handling samples.

(e) Lists of major analytical instruments and support equipment.

(f) Procedures for calibration, verification and maintenance of major analytical instruments and support equipment.

(g) Procedures for evaluating quality control samples, including, but not limited to, method blanks, laboratory control samples, matrix fortified samples and replicates.

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

(i) Procedures for reviewing analytical data and reporting analytical results.

(4) REVISIONS. The quality manual shall be kept current by the responsible party, however named, for maintaining the laboratory's quality system. All editions or versions of the quality manual shall indicate the dates in which they were issued or revised.

(5) LABORATORIES ANALYZING DRINKING WATER SAMPLES. Laboratories performing tests in drinking water shall ensure, in addition to the requirements in this section, that the content elements specified in Chapter III of the "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA 815−R−05−004, fifth edition, EPA, Office of Groundwater and Drinking Water, January 2005, are addressed, included, or referenced in their quality manuals.

Note: This document is available at: http://www.epa.gov/safewater/labcert/labindex.html.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.38 Corrective action for quality system and quality control samples. (1) The laboratory shall take corrective action when:

(a) Departures from established policies and procedures in the quality system are identified or become apparent.

(b) Quality control samples, including proficiency testing samples, fail established acceptance limits or evaluation criteria.

(2) The corrective action shall identify the source of the problem, correct the problem, and have a mechanism to verify the action has had the desired effect.

(3) The laboratory shall document corrective action taken to address the nonconformance and any other changes resulting from corrective action investigations. Changes taken to address failures of quality control samples to meet established acceptance criteria shall be those that resolve or address the failure in an expeditious manner before affected results are released or reported by a laboratory.

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

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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 that are part of its quality system and that are required to demonstrate compliance with this chapter for a minimum of 3 years after the generation of the last entry in a record or document. The laboratory shall retain records and documents for a longer minimum period, if they are necessary to reconstruct analytical results generated during a 3−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 certification or registration 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 certified or registered.

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

(g) Records and documents shall be legible and their entries shall be safeguarded against obliteration, erasures, overwriting and corruption.

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 their 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. Administrative records that laboratories shall maintain include:

(a) Certificates of certification or registration issued by the department, unless the department has requested a laboratory to return them.

(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 certified or registered 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) Records of demonstration of capability for each analyst required to perform the demonstrations specified in s. NR 149.36 (3).

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

(3) ANALYTICAL AND TECHNICAL RECORDS. (a) 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.

(b) 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 and forms, in hard copy or electronic media.

(c) Analytical and technical records retained by the laboratory shall allow access to information that includes:

1. Collection, arrival, processing and analysis dates of samples received for analysis.

2. Collection and analysis time for tests with holding time of 48 hours or less.

3. Preservation status of samples on arrival at the laboratory.

4. Identity of laboratory personnel preparing and testing samples.

5. Identification of the analytes or analyte groups analyzed in samples.

6. Preparatory techniques, such as digestions, extractions and clean−ups, to which samples are submitted.

7. Methods of analysis used for samples.

8. Results of sample analysis.

9. Traceability of standards and reagents used to perform analysis.

10. Calibration verification information and measurements of laboratory support equipment associated with sample analysis and storage.

11. Initial and continuing calibration data associated with samples analyzed.

12. Raw data for analytical instrument calibrations and samples. The department has exempted the retention of emission counts for samples and standards analyzed after an initial calibration for older models of inductively coupled plasma emission spectrophotometers that are incapable of providing that information when operated in the instrument calibration mode.

13. Results of quality control samples associated with samples analyzed.

14. Corrective actions associated with samples analyzed.

15. Maintenance performed on laboratory support equipment and analytical instruments.

16. Environmental conditions crucial to tests performed at laboratory facilities at the time samples are analyzed.

17. Reports of final results submitted to clients or the department.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.40 Standard operating procedures. **(1)** GENERAL REQUIREMENTS. (a) Laboratories shall maintain written standard operating procedures that document or reference activities needed to maintain their quality systems and that enable performing or reproducing an analysis in its entirety as performed at the laboratory.

(b) 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.

(c) Standard operating procedures may consist in part of copies of published documents, manuals or procedures if:

1. Modifications to the published source are described in writing in additional documents.

2. 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.

(d) Standard operating procedures shall indicate their dates of issue or revision.

(2) ANALYTICAL METHODS MANUAL. (a) The laboratory shall have and maintain a list describing analytical test methods performed for programs covered by this chapter.

(b) The analytical methods manual may consist of published or referenced test methods, or standard operating procedures written by the laboratory as allowed in this section.

(c) The essential elements of test methods required in par. (d) may be presented in narrative, tabular, schematic or graphical form. The analytical methods manual shall be an identifiable document in hard copy or electronic format traceable to the laboratory.

(d) When the analytical methods manual consists of standard operating procedures written by the laboratory, each standard operating procedure shall include, address or refer to, at a minimum, the following elements:

1. Identification of the test method.
2. Applicable analytes.
3. Applicable matrices.
4. Method sensitivity.
5. Potential interferences.
6. Equipment and analytical instruments.
7. Consumable supplies, reagents and standards.
8. Sample preservation, storage and hold time.
9. Quality control samples and frequency of their analysis.
10. Calibration and standardization.
11. Procedure for analysis.
12. Data assessment and acceptance criteria for quality control measures.
13. Corrective actions and contingencies for handling out of control or unacceptable data.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.41 Method selection. **(1)** The laboratory shall use methods for environmental testing approved 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.

(2) When methods are not prescribed by covered programs under this chapter or permits issued by the department, the laboratory shall consult with the department to select a method that is suitable for the matrix, type of analyte, expected level of analyte, regulatory limit and anticipated interferences in the sample.

Note: A list of authoritative sources for methods and quality control information is provided in Appendix III to this chapter.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.42 Alternative methods. **(1)** The department may allow the use of alternative methods from those prescribed by programs covered under this chapter, including the safe drinking water program, if a laboratory requests approval and if the environmental protection agency 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 specified by programs covered under this chapter, for any of the following situations:

(a) The EPA has granted approval for the alternative methods.

(b) The applicable covered program, after consultation with the laboratory certification and registration program, determines that the allowance does not result in a detrimental effect on the quality and defensibility of the results to be generated.

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

(3) The request for consideration of approval for use of 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 test 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 test 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 laboratory certification and registration program 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.

Note: A list of authoritative sources for test methods is provided in Appendix III of this chapter.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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

(a) 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 this is required by approved test methods or when they influence the quality of test results.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

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

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

(2) LABORATORY SUPPORT EQUIPMENT. (a) All support equipment shall be kept in working order by submitting it to 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 satisfactory before the equipment is returned to service.

(3) CALIBRATION AND VERIFICATION OF SUPPORT EQUIPMENT. (a) All support equipment shall be calibrated or verified over its range of use using available reference materials traceable to NIST. When reference materials traceable to NIST are not produced, manufactured or 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 verification checks shall be established by approved test methods, department guidance, or in their absence, tolerances established by manufacturers.

Note: Department guidance documents are available for download from the department website at: http://www.dnr.state.wi.us/org/es/science/lc/OUTREACH/Guidance.htm.

(c) When the results of the calibration or calibration verification of support equipment do not meet the specifications of the application or method for which the equipment is used, the equipment shall be removed from service until repaired; however, if the deviation from the calibration specifications results in a consistent bias, the equipment may remain in service if correction factors are applied to all measurements made with the deviating equipment.

(d) Devices used to measure the temperature of laboratory ovens, incubators, water baths, refrigerators, freezers and samples received at the laboratory shall be calibrated or verified at least yearly against thermometers traceable to NIST.

(e) The operating temperature of autoclaves, incubators, ovens and water baths used as part of a method shall be checked to meet the temperature requirements of that method each day they are used.

(f) Refrigerators, freezers, ovens and incubators holding samples continuously as part of standard operating conditions shall be checked on each day that laboratory personnel conduct analyses. The laboratory shall endeavor to set equipment settings and conditions that maintain required temperatures on days that personnel do not conduct analyses.

(g) Analytical balances that have been used at least once in a month shall be checked monthly with at least 2 certified weights, one weight in the gram range and one weight in the milligram range. The weights used to perform these checks shall be:

1. Traceable to NIST, and shall be of class or type suitable for verifying the accuracy of analytical balances.

2. Certified for accuracy every 5 years by a metrology service outside the laboratory or new individual weights of suitable class or type traceable to NIST shall be purchased for use. This re−certification shall be performed sooner than every 5 years if balance checks performed using these weights suggest that a change in the certified weight has occurred.

3. Handled and stored in a manner that protects their integrity.

(h) Non−analytical balances that have been used at least once in a month shall be checked monthly with at least one weight in the expected range of their use. The weights used to perform these checks may be traceable to or verified against those traceable to the NIST.

(i) Mechanical and automatic volumetric dispensing devices, including pipettes, micro−pipettes, burettes and automatic dilutors and dispensers shall be checked for accuracy at least quarterly when they are in use.

1. Glass microliter syringes do not need to be checked for accuracy if they are documented to be as accurate as class A glassware.

2. Disposable pipettes and any of the aforementioned devices which are dedicated to use in method steps or applications that do not require use of class A glassware are exempted from the quarterly verification of accuracy.

(4) LABORATORY ANALYTICAL INSTRUMENTS. (a) Laboratory analytical instruments shall be operated by personnel trained in their use. Instructions on the use and maintenance of equipment shall be available to instrument operators.

(b) All instruments shall be properly maintained, inspected and cleaned. The laboratory shall establish procedures for the maintenance of analytical instruments to prevent contamination or deterioration that may affect reported results.

(c) Analytical instruments that give suspect results or that have been shown to be defective or outside of performance specifications shall be taken out of service.

(d) When analytical instruments leave the direct control of the laboratory for maintenance or for any other reason, the laboratory shall ensure that the functional and calibration status of those analytical instruments are checked or demonstrated to be satisfactory before the instruments are returned to service.

(5) INSTRUMENT CALIBRATION GENERAL PROVISIONS AND REQUIREMENTS. (a) All analytical instruments shall be calibrated at least once in any year in which they have been used, and shall be calibrated or their calibration verified before they are used to provide any quantitative results.

(b) When more stringent instrument initial calibration or continuing calibration verification requirements are required in mandated test methods or regulations, laboratories shall follow the more stringent requirements, unless:

1. A test method requires analyzing more than 3 standards to establish a linear calibration, and the laboratory chooses to narrow the calibration range of the determination to no more than 2 orders of magnitude and uses at least 3 standards to generate an initial calibration.

2. A test method requires analyzing more than one continuing calibration verification standard to verify a linear calibration and the laboratory has narrowed the calibration range of the determination to no more than 2 orders of magnitude and uses at least one standard to verify continued calibration.

(6) INITIAL INSTRUMENT CALIBRATION. (a) The details of initial instrument calibration procedures, including, calculations, integrations, acceptance criteria and associated statistics shall be included or referenced in the test method standard operating procedure. When initial instrument calibration procedures are cited by reference in the test method standard operating procedure, the laboratory shall retain the referenced material.

(b) The laboratory shall select a calibration model that is appropriate for the expected behavior of the analytical instrument to be calibrated.

(c) To establish calibration, the laboratory shall select a number of non−zero standard concentrations that is appropriate for the calibration model selected and the expected range of concentrations. The number of calibration standards used shall also be sufficient to establish a relationship or corroborate a universally established theoretical relationship between instrument response and concentration that is appropriate for the specific instrument and its intended use.

(d) The minimum number of standard concentrations selected to establish calibration shall be 3 except for:

1. Dissolved oxygen meters, which shall be calibrated against water−saturated air, air−saturated water at a known temperature and pressure, or by reference to an aliquot of air−saturated water analyzed by the Winkler or iodometric method.

2. Ion selective electrodes and pH meters, the minimum number shall be 2.

3. Inductively coupled plasma emission spectrophotometers and inductively coupled plasma mass spectrometers, the minimum number shall be one.

4. Calibration models that are quadratic, the minimum shall be 5.

5. Calibration models that are cubic, the minimum shall be 7.

(e) 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 to be quantitated with an initial calibration. Laboratories reporting results at levels at or

near the limit of detection of an analysis shall include in initial calibrations a standard at a concentration near the limit of quantitation of the analysis.

(f) To generate a calibration function, the laboratory shall select a reduction technique or algorithm that is appropriate for the calibration model and number of standard concentrations selected.

1. 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 fluid 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 pH meters 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.

2. The laboratory shall use the simplest linear calibration function unless it has documentation that a non−linear function provides a statistically improved definition of the calibration range. Non−linear functions may not be used to compensate for instrument saturation, insensitivity, or malfunction.

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

4. The laboratory may not use reiterative reduction techniques or algorithms that force calibration functions through zero.

Note: Reiterative reduction techniques or algorithms that force through zero obtain mathematically, by repeated application, a null response for a zero standard that has a non−zero 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 response factors or automatic zeroing as part of an initial calibration, when methods, regulations or covered programs allow those techniques.

(g) 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.

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

2. When linear regression or least squares analysis is used to reduce calibration data for inorganic analytes and metals, the correlation coefficient of the resultant calibration curves shall be at least 0.995.

3. When linear regression or least squares analysis is used to reduce calibration data for organic analytes, the correlation coefficient of the resultant calibration curves shall be at least 0.99.

4. When quadratic regression analysis is used to reduce calibration data for inorganic analytes and metals, the coefficient of determination of the resultant calibration curves shall be at least 0.995.

5. When quadratic regression analysis is used to reduce calibration data for organic analytes, the coefficient of determination of the resultant calibration curves shall be at least 0.99.

(h) The laboratory shall establish procedures for zeroing an instrument and the treatment of calibration blanks, when the referenced analytical method used by the laboratory requires the response of a calibration blank to be part of a calibration function.

(i) Laboratories shall verify all initial instrument calibrations after they are generated but before they are used to quantitate any samples, with a second source standard, unless either of the following conditions exists:

1. An instrument is calibrated by tuning it to conform to a universally accepted scientific law or scale, as is the case with pH meters, ion selective electrodes and dissolved oxygen meters.

2. The laboratory analyzes quality control standards for the analyte or analyte group involved and evaluates them as specified in s. NR 149.48 (5).

(j) Unless otherwise required by regulation, method or program, the acceptance criteria for this second source verification shall be that required under sub. (7) for continuing instrument calibration verification.

(k) Laboratories shall quantitate sample results only from initial instrument calibrations, unless otherwise allowed by regulation, method or covered program.

(L) Laboratories shall quantitate sample results from an instrument response that is within the range of the initial calibration. If sample dilution is required, the dilution shall be the lowest required to obtain an instrument response within the range of the initial calibration.

1. Except for samples analyzed by inductively coupled plasma emission spectrophotometers and inductively coupled plasma mass spectrometers, samples having responses greater than that of the most concentrated standard of an initial calibration allowed to be established with at least 3 different standard concentrations shall be diluted and reanalyzed. When samples cannot be diluted and reanalyzed, sample results shall be reported with appropriate qualifiers or narrative warnings.

2. Samples analyzed by inductively coupled plasma emission spectrophotometers and inductively coupled plasma mass spectrometers having responses at or above 90% of the established upper limit of the linear dynamic range of the instruments shall be diluted and reanalyzed. When samples cannot be diluted and reanalyzed, sample results shall be reported with appropriate qualifiers or narrative warnings.

3. Samples analyzed by inductively coupled plasma emission spectrophotometers and inductively coupled plasma mass spectrometers having responses below 90% of the established upper limit of the liner dynamic range of the instruments but above the response of the highest concentration of standard in an initial calibration may be reported without resorting to dilution.

(m) 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.

(n) Laboratories shall perform an initial calibration after instruments undergo non−routine maintenance, when repeated use or other conditions change their expected behavior, and when their continuing calibration cannot be verified.

(o) Except as allowed in s. NR 149.39 (3) (c) 12., laboratories shall retain all the raw data necessary to reconstruct or reproduce, independently of analytical instruments, all calibration functions associated with initial calibrations.

(7) CONTINUING INSTRUMENT CALIBRATION VERIFICATION. (a) When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to quantitating samples by continuing calibration verification with each analytical batch and at least once on each analysis day. Continuing calibration verification shall also be performed after the consecutive analysis of each group of 20 samples, if 20 or more samples constitute an analytical run. Continuing calibration verification is not required for analyses that cannot be spiked, such as BOD, cBOD and TSS, or those analyses that do not involve a calibration, such as titrations.

(b) The calibration standards analyzed to demonstrate continuing instrument calibration may be obtained from the same source used to generate an initial calibration.

(c) The number and concentration of calibration standards required to demonstrate continuing instrument calibration is outlined in Table 1 of this subchapter.

Table 1
Requirements for Continuing Calibration Verification

Calibration Function	# of Standards Required for Verification	Concentration of Verification Standard
Tuning an instrument to conform to a universally accepted scientific law or scale (i.e. electrometric techniques)	The laboratory shall analyze at least a single verification standard	The concentration of the standard shall be within the range established during the initial calibration
Average response/ calibration factor, linear regression, least squares analysis, or otherwise obeys a linear model	The laboratory shall analyze at least a single verification standard	The concentration of the standard may be varied within the established calibration range.
Quadratic regression, 2nd order polynomial, or other quadratic model	The laboratory shall analyze at least 2 verification standards	One of the standard concentrations shall be chosen to verify continuing calibration near the point of inflection of the calibration function
Cubic regression, 3rd order polynomial, or other cubic model	The laboratory shall analyze at least 3 verification standards	Two of the standard concentrations shall be chosen to verify continuing calibration near the points of inflection of the calibration function
Discrete or non−smooth segments	The laboratory shall analyze one standard per calibration segment	The concentrations of the standards shall be different from the ones used to establish each segment.

(d) The acceptance criteria for continuing calibration verification standards shall be those defined in the method utilized by the laboratory. If the reference method does not contain criteria, the acceptance criteria for continuing calibration shall be:

1. Obtaining concentrations within 10% of the respective actual concentrations of all reportable inorganic analytes and metals from an initial calibration.

2. Obtaining concentrations within 15% of the respective actual concentrations of all reportable organic analytes from an initial calibration.

(e) When the continuing calibration verification results obtained are outside acceptance criteria, the laboratory shall perform another calibration verification if the results of this second calibration verification fail to meet acceptance criteria, the laboratory shall take corrective action. After taking corrective action, the laboratory shall perform 2 consecutive calibration verifications that meet acceptance criteria or shall perform another initial calibration.

(f) Samples associated with a failing calibration verification shall be reanalyzed or reported with appropriate qualifiers.

(g) The details of the continuing instrument calibration procedure, calculations and associated statistics shall be included in the test method standard operating procedure. When continuing calibration verification procedures are cited by reference in the test method standard operating procedure, the laboratory shall retain the referenced material.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.45 Measurement traceability. (1) STANDARDS, REAGENTS AND REFERENCE MATERIALS. (a) The laboratory shall ensure that results of analyses can be linked to all the standards and reagents used to derive results. Standards and reagents used in analyses shall conform to the purity specifications contained in approved methods of analysis. When approved methods of analysis do not specify the purity of the standards and reagents to be used, the laboratory shall choose standards and reagents of sufficient purity to ensure the validity of reported results.

(b) The laboratory shall certify the accuracy of all reference materials used to calibrate or verify the calibration of analytical support equipment. Reference materials shall be calibrated by a body independent of that in charge of analytical operations that can provide traceability to primary standards maintained by NIST. When reference materials traceable to NIST are not produced, manufactured or 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.

(c) The laboratory may not use standards and reagents beyond their expiration dates, unless the laboratory can verify their reliability in a defensible manner.

(2) DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS AND REFERENCE MATERIALS. (a) The laboratory shall document the identity, source and purity of all standards and reagents used in tests methods performed. The laboratory shall retain records of certificates of analysis or purity, when the records are provided by the supplier, and are necessary to establish the identity, source or purity of standards and reagents.

1. Original containers of standards and reagents shall be labeled with a receipt and an expiration date.

2. The laboratory shall document the lot number, manufacturer, date of receipt and the date of expiration of stock standards and reagents separately from their containers to ensure this information will be retained when the containers are discarded.

3. The laboratory shall maintain records that detail the preparation of intermediate and working standards and reagents. These records shall link the intermediate and working standards and reagents to their respective originating stocks or neat compounds and shall indicate their date of preparation, expiration and the identity of the preparer.

(b) The laboratory shall retain records and certificates that trace reference materials used to calibrate or verify analytical support equipment to the source of the corresponding reference materials. The laboratory shall retain records demonstrating that the accuracy of the reference materials has been certified or verified, at the required frequencies, by a body outside of that in charge of analytical operations.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.46 Handling of samples. (1) SAMPLE COLLECTION. (a) The laboratory shall retain records supplied by the collector to allow the laboratory to evaluate collection procedures against the laboratory's sample acceptance policy.

(b) When the laboratory provides containers and preservatives for sample collection, including bulk sampling containers such as "carboys", the laboratory shall have standard operating proce-

dures in place which address concerns that the containers are adequately cleaned and not contributing to contamination of samples, do not contain analytes of interest at levels which will affect sample determinations and that the preservatives used are sufficiently pure to maintain the validity of reported results. Containers supplied by the laboratory for sample collection shall allow collecting a sufficient amount of sample to perform all required or requested determinations at the required or desired sensitivity.

Note: The laboratory should establish procedures to ensure and document that the sample containers it provides do not contribute contaminants before they are used for collecting samples.

(2) SAMPLE ACCEPTANCE POLICY. (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.

1. Drinking water samples received beyond holding time, improperly preserved, in inappropriate containers or showing evidence that they have not been collected according to approved or accepted protocols shall be rejected for analysis, unless the laboratory can document that it has been instructed by the client to proceed with analyses, and all associated results are accompanied by a disclaimer attesting that results may not be used to determine or evaluate compliance with the safe drinking water act.

2. The results of samples that are not drinking waters shall be appropriately qualified if the samples are received improperly preserved, in inappropriate containers, beyond holding time, with insufficient volume to complete requested analyses, or if the laboratory has evidence that the samples have not been collected according to approved or accepted protocols. Alternatively, the laboratory may reject the samples for analysis.

(b) When samples received do not conform to the descriptions provided by a collector, the laboratory shall consult with the collector or sample originator to determine the processing or disposition of the samples.

(3) SAMPLE HANDLING PROTOCOLS. (a) The laboratory shall establish and follow procedures for identifying samples uniquely. The procedures shall ensure that the identity of samples cannot be confused physically or when referenced in records or other documents.

1. Samples received by a laboratory for analysis shall be assigned a unique identification code.

2. The unique identification code shall be placed on a sample container as a durable label.

3. The unique identification code shall be used as a link to associate samples with their complete history, including treatment and analysis, while in the laboratory's possession.

(b) Chain−of−custody documentation shall be required for those facilities that do not perform their own sample collection, transport and analysis.

(c) The laboratory shall apply evidentiary chain of custody procedures when it receives samples that support regulatory investigations or when required to do so in accordance with a written agreement between the laboratory and the client.

(4) SAMPLE PRESERVATION AND HOLDING TIME. (a) Laboratories shall follow the sample preservation procedures and holding times required by state and federal regulations. If the sample preservation procedures and holding times are not required by state or federal regulations, laboratories shall follow the sample preservation procedures and holding times established in the analytical method. If the analytical method does not establish sample preservation procedures or holding times, laboratories shall follow the procedures in the authoritative sources specified in Appendix III of this chapter.

Note: Sample preservation procedures and holding times are given in 40 CFR 136, ch. NR 219, SW–836 "Test Methods for Evaluating Solid Waste" as cited in item 24 of Appendix III of this chapter, and may be specified in the analytical methods.

(b) Samples requiring preservation at 6ºC under this section shall be considered preserved if they are received at a temperature from above their freezing point to 6ºC or if they are received surrounded by ice. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

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.

Note: The preservation status of samples may be recorded as "received on ice" only if solid ice is present around samples when they are received at the laboratory. The preservation status of samples refrigerated with ice packs, such as "blue ice", should not be recorded as "received on ice".

(c) When multiple samples requiring thermal preservation at 6ºC are received in the same cooler or holding container, the entire set of samples shall be considered preserved if the temperature of a blank or a sample is determined to be from above freezing to 6ºC, or if there is ice remaining in the shipment container.

(d) Samples to be analyzed for whole effluent toxicity shall be considered preserved if their temperature on receipt is above freezing and does not exceed 10ºC.

(e) Except as specified in pars. (b) to (d), samples requiring thermal preservation at a temperature other than 6ºC shall be considered preserved if their temperature on receipt is within plus or minus 2 degrees of the required preservation temperature.

(5) 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 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 and laboratory receipt.

(c) The times of sample collection and laboratory receipt for samples to be analyzed for tests with holding times equal to or less than 48 hours.

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

(e) Documentation of sample preservation status and other sample conditions on receipt.

(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 them.

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

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

(b) Samples requiring thermal preservation at temperatures other than 6ºC shall be stored under refrigeration within 2 degrees of the specified preservation temperature.

(c) Samples requiring thermal preservations at 6ºC may be stored at temperatures from above their freezing point to 6ºC.

(d) Samples shall be stored separately from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in areas that prevent or minimize cross−contamination.

(e) Sample extracts, digestates, leachates or concentrates, resulting from any initial preparatory step, shall be stored as specified in this subsection.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.47 Laboratory test reports. (1) GENERAL PROVISIONS, FORMAT AND CONTENT. (a) The results of each test performed by a laboratory shall be reported in accordance with any

requirements or instructions specified in approved methods or by the department.

(b) Laboratory test reports shall have formats that facilitate conveying or reviewing the content elements specified in this section, unless otherwise provided by pars. (c), (d) and (e). Content elements may be presented in narrative, tabular, schematic or graphical form, in hard copy or electronic media.

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

(d) Laboratories that are operated by a facility whose function is to provide data to monitor the facility's compliance with department programs covered by this chapter 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:

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.

(e) Unless otherwise specified by department programs that receive data on behalf of facilities, directly from laboratories, or when provided by pars. (c) and (d), test reports from laboratories shall include at least the following information.

1. The name, address and telephone of the laboratory where tests were performed, as well as the name of a contact.

2. The laboratory's certification or registration identification number.

3. The name and address of the client or entity whose samples were analyzed.

4. The sample codes or identifiers provided by the client or collector.

5. Identification of or reference to the methods used for analysis.

6. The collection date of the samples.

7. The date of receipt of the samples.

8. For samples submitted to pretreatment steps, such as digestions or extractions, with identified holding times in department regulations, the date in which such steps were performed.

9. The date of analysis.

10. Results of analyses with their respective measurement and reporting units.

a. For sample results requiring adjustment for dilutions, the dilution factors.

b. For sample results reported on a dry weight basis, the solids content and a statement or flag indicating that results have been adjusted for the solids content of the corresponding samples.

11. For tests for which the department requires reporting to the limit of detection, the limits of detection and quantitation of the associated results.

a. For sample results requiring adjustment for dilutions, an indication of whether the detection and quantitation limits have been adjusted for the corresponding sample dilutions.

b. For sample results reported on a dry weight basis, an indication of whether the detection and quantitation limits have been adjusted for the solids content of the corresponding samples.

12. The names and signatures of responsible parties authorizing reported results.

13. Descriptions of any deviations encountered by the laboratory from chapter requirements or procedures referenced in approved methods, when the deviations affect the validity or the defensibility of reported results.

a. Description of these deviations may be communicated through narratives, flags or qualifiers.

b. When flags or qualifiers are used to declare these deviations, the laboratory shall include a key to describe the meaning of all used flags and qualifiers.

14. The date of the test report.

(2) AMENDMENTS TO LABORATORY TEST REPORTS. (a) Amendments to test reports already issued by a laboratory shall be made by an authorized laboratory representative 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.

(3) TEST RESULTS OBTAINED FROM SUBCONTRACTORS. (a) When reports contain results of tests performed by subcontractors, the associated results shall include any qualifiers noted by the subcontract laboratory and shall be identified with the subcontractors' facility identification codes.

(b) Subcontractors shall provide upon request of the originating laboratory or the department all the information contained in this section.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.48 Quality control requirements for chemical testing. (1) GENERAL REQUIREMENTS. (a) Laboratories shall establish a quality control program that includes the analysis of appropriate samples, such as method blanks, laboratory control samples, matrix spikes, matrix spike duplicates, replicates, surrogate spikes and analytical protocols, such as detection limit studies and confirmatory techniques. These quality control procedures shall be used to assess:

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) At least annually, laboratories shall review and evaluate the acceptability criteria specified in this section for all quality control samples and measures, and update the criteria whenever the performance characteristics of any of these samples and measures change.

(c) Laboratories may not adjust or correct the sample results by the recoveries of associated laboratory control samples, matrix spikes and surrogates, unless a method or project plan approved by the department requires it. Laboratories may not subtract analyte concentrations found in method blanks from sample results unless a method or project plan approved by the department requires it.

(d) Laboratories shall establish procedures for identifying and documenting preparation batches that facilitate determining compliance with the frequencies of quality control samples required by this subchapter.

(2) LIMITS OF DETECTION AND QUANTITATION. (a) Laboratories shall determine the limit of detection for all tests performed and for all analytes reported except for:

1. Biochemical oxygen demand.

2. Tests for which analyzing a fortified sample is impossible.

3. Titrimetric tests.

4. Gravimetric tests, other than oil and grease as hexane extractable materials.

(b) Laboratories shall determine the limit of detection of an analyte by a protocol established by regulation or as referenced in

approved methods of analysis. All sample−processing steps of a test method shall be included in the determination of a limit of detection.

(c) For tests for which this chapter does not require performing a limit of detection, laboratories shall establish estimates of a test's sensitivity based on the intended use of the data for a given application.

(d) Limits of detection shall be determined at least annually unless a laboratory can verify the continued applicability of a previously determined limit of detection by an established and defensible protocol.

(e) Limits of detection shall be determined each time there is a change in a test method or instrumentation that affects the sensitivity of an analysis.

(f) Laboratories shall establish procedures to relate limits of detection to limits of quantitation.

(g) Established limits of quantitation shall be above determined limits of detection.

(3) METHOD BLANK. (a) Method blanks shall be processed 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 appropriate or required for analysis of pH, alkalinity, conductivity and solids determinations.

(b) Method blanks shall be processed at a frequency of at least one per preparation batch. When samples are analyzed by methods that do not require a preparation step before analysis, a blank, different from a calibration blank, shall be analyzed at the frequency of one per analytical batch.

(c) Whenever a method blank contains analytes of interest above the detection limit of an analysis, the laboratory shall evaluate the nature of the interference and its effect on each sample in a preparation batch.

(d) A sample in a batch shall be reanalyzed or qualified if the concentration of an analyte of interest in the associated method blank exceeds the highest of any of the following values:

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

(4) LABORATORY CONTROL SAMPLES. (a) Unless otherwise exempted by this subsection, a laboratory control sample shall be processed at a frequency of at least one sample per preparation batch, along with and under the same conditions as the associated samples in a preparation batch. These conditions shall include all sample preparation steps, except waste characteristic extractions.

Note: Waste characteristic samples are fortified after the extraction is completed.

(b) Laboratory control samples for the biochemical oxygen demand and carbonaceous biochemical oxygen demand tests shall be fortified with a mixture of glucose and glutamic acid as specified in approved methods of analysis. These laboratory control samples shall be processed at a frequency of at least one sample per analytical batch for laboratories that analyze more than 20 samples per week. Laboratories that analyze fewer than 20 samples per week shall analyze, at a minimum, one laboratory control sample per week.

(c) Laboratory control samples are not required to be processed for tests for which analyzing a fortified sample is impossible or impractical, or when a laboratory follows par. (e).

Note: Laboratory control samples need not be analyzed for the following tests: pH, solids determinations, chlorophyll a, color, odor, oil and grease as freon extractable material.

(d) Matrix spikes or certified reference materials may be processed for all reported analytes, at the frequency described in par. (a), in place of laboratory control samples, if the acceptance criteria for corresponding laboratory control samples are used to evaluate the matrix spikes and the laboratory takes the corrective action required in this subsection when matrix spikes fail established laboratory control sample acceptance criteria.

(e) For analyses of polychlorinated biphenyls, the laboratory shall fortify a laboratory control sample 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 a laboratory control sample 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 laboratory control samples at least once every year that any of those analytes are reported.

(f) The laboratory shall compute the recovery of each fortified analyte in a laboratory quality control sample. The laboratory shall evaluate the results of laboratory control samples against acceptance criteria published by the department, or when the department has not published acceptance criteria, against:

1. Criteria contained in approved methods of analysis.
2. Laboratory generated acceptance criteria when approved methods of analysis do not contain acceptance criteria.
3. Criteria specified in project quality plans approved by the department.

(g) When laboratory control samples do not meet acceptance criteria, the laboratory shall reprocess or reanalyze all samples associated with the failing laboratory control samples or qualify the results of all samples in the preparation batch.

(h) Laboratories may process and analyze replicate laboratory control samples to establish a measure of the ability of an analytical system, independent of matrix effects, to reproduce results. The laboratory may reprocess or reanalyze all samples, or qualify the results of all samples in a preparation batch, if the relative percent difference of laboratory sample control duplicates exceeds criteria established by the laboratory.

(5) QUALITY CONTROL STANDARDS. (a) Laboratories that do not use second source standards to verify the accuracy of initial calibrations shall analyze quality control standards as defined in s. NR 149.03 (57), 3 times per year at evenly spaced intervals for all certified or registered analytes determined by tests amenable to fortification, and for which known quality control samples are commercially available.

Note: Analysis of quality control standards is not required for tests, such as pH, which are performed using instruments calibrated by tuning them to conform to a universally accepted scientific law or scale. These tests are also exempt from initial calibration verification with a second source standard.

(b) Laboratories shall evaluate the results of known quality control samples against the acceptance criteria supplied by the provider. If the results of known quality control samples exceed the acceptance limits issued by a provider, the laboratory shall take corrective action and demonstrate within 30 days, through analysis of another known quality control sample or processed second source standard, the effectiveness of the corrective action taken.

(6) MATRIX SPIKES AND MATRIX SPIKE DUPLICATES. (a) Matrix spikes and matrix spike duplicates corresponding to the quality system matrix to which collected samples are assigned shall be processed and analyzed, unless as allowed in sub. (7) (a), when:

1. Mandated test methods require their analysis and a sufficient volume or amount of sample has been received to permit their analysis.
2. Project plans require their analysis.
3. They are used in place of laboratory control samples to evaluate the level of control of an analytical system.

Note: Matrix spikes need not be analyzed for the following tests: biochemical oxygen demand, carbonaceous biochemical oxygen demand, pH, solids determinations, alkalinity, acidity, chlorophyll a, color, odor, oil and grease as freon extractable material.

(b) When required to be analyzed by par. (a), matrix spikes and matrix spike duplicates shall be:

1. Processed along with and under the same conditions as the associated samples in a preparation batch. These conditions shall include all sample preparation steps, except waste characteristic extractions.

Note: Waste characteristic samples are fortified after the extraction is completed.

2. Processed and analyzed at a frequency of one per preparation batch of samples consisting of the same quality system matrix or at frequency specified by a project plan or client agreement.

3. Fortified with the analytes specified in approved methods, project plans, client agreements or with all reported analytes, except as allowed in sub. (4) (e).

4. Fortified with all reported analytes when matrix spikes are used in place of laboratory control samples.

(c) The laboratory shall compute the recovery of each fortified analyte in a matrix spike and matrix spike duplicate, and the relative percent difference or absolute difference of each fortified analyte in a matrix spike and matrix spike duplicate pair. The laboratory shall evaluate the recoveries, and the relative percent difference or absolute range against acceptance criteria published by the department, or when the department has not published criteria, against:

1. Criteria contained in approved methods of analysis.

2. Laboratory generated acceptance criteria when approved methods of analysis do not contain acceptance criteria.

3. Criteria specified in documented and approved project quality plans, or client agreements.

(d) When matrix spikes or matrix spike duplicates do not meet acceptance criteria, the laboratory shall reprocess, reanalyze or qualify the results of the chosen fortified sample in the preparation batch. When the laboratory determines that the failure of matrix spikes or matrix spike duplicates has affected other samples in the same preparation batch, the laboratory shall reprocess or reanalyze the samples, or qualify their results.

(7) SAMPLE REPLICATES. (a) Sample replicates may be analyzed in place of matrix spike duplicates when there is a high probability that a replicate pair will contain the analytes of interest at or above the limit of quantitation of an analysis.

(b) Sample replicates corresponding to the quality system matrix to which collected samples are assigned, shall be processed and analyzed when:

1. Mandated test methods require their analysis and a sufficient volume or amount of sample has been collected or received to permit their analysis.

2. Project plans require their analysis.

3. Clients, by agreement with a laboratory, require their analysis.

(c) When required to be analyzed by par. (b), sample replicates shall be:

1. Processed along with and under the same conditions, including all sample preparation steps, as the associated samples in a preparation batch.

2. Processed and analyzed at a frequency of one per preparation batch of samples consisting of the same quality system matrix or at a frequency specified by a project plan or client agreement.

(d) The laboratory shall compute the relative percent difference or absolute difference of each pair of sample replicates. The laboratory shall evaluate these results against acceptance criteria published by the department, or when the department has not published acceptance criteria, against:

1. Criteria contained in approved methods of analysis.

2. Laboratory generated acceptance criteria when approved methods of analysis do not contain criteria.

3. Criteria specified in documented and approved project quality plans or client agreements.

(e) When sample replicates do not meet acceptance criteria, the laboratory shall reprocess, reanalyze or qualify the results of the chosen sample analyzed in replicate in the preparation batch. When the laboratory determines that the failure of sample replicates has affected other samples in the same preparation batch, the laboratory shall reprocess or reanalyze the samples or qualify their results.

(8) SURROGATE SPIKES. (a) Surrogate compounds specified in approved methods of analysis or documented and approved project plans shall be added to all samples in a preparation batch, including method blanks, laboratory control samples, matrix spikes, matrix spike duplicates and replicates.

(b) The laboratory shall compute the recovery of all surrogates added to each sample in a preparation batch. The laboratory shall evaluate these results against acceptance criteria published by the department, or when the department has not published acceptance criteria, against:

1. Criteria contained in approved methods of analysis.

2. Laboratory generated acceptance criteria when approved methods of analysis do not contain criteria.

3. Criteria specified in documented and approved project quality plans or client agreements.

(c) When surrogate recoveries do not meet acceptance criteria, the laboratory shall determine whether the failures are the result of matrix interference. If the failures result from matrix interference, the laboratory shall qualify the results of the affected samples. If the failures cannot be attributed to matrix interference, the laboratory shall reprocess and reanalyze the affected samples or qualify sample results.

(9) SELECTIVITY. (a) The laboratory shall establish procedures to confirm the results of organic analytes determined by techniques that, unlike mass spectrometry, do not provide a positive unique identification when:

1. The history of a sample source does not suggest the likely presence of the detected analyte.

2. A client or approved project plan requires it.

(b) The laboratory shall establish procedures and rules for reporting results for samples analyzed by dual column and dual detector systems that declare:

1. Under what conditions a presumptive identification is confirmed.

2. Under what conditions a presumptive identification is reported.

3. The value that will be reported when the dual systems both provide quantitative confirmed results.

(c) The laboratory shall develop and document acceptance criteria, for chromatographic retention time windows, which consider retention time shifts due to routine column maintenance.

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

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.

NR 149.49 Quality control requirements for 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", 2nd edition.

Note: The referenced manual can be obtained at http://dnr.wi.gov/org/water/wm/ww/biomon/

(2) CHEMICAL TESTING IN SUPPORT OF WHOLE EFFLUENT TOXICITY TESTING. Laboratories performing tests for alkalinity, ammonia, hardness, pH, conductivity, dissolved oxygen and total residual chlorine shall follow the quality control requirements specified in s. NR 149.48 except that laboratories need not analyze matrix spikes or matrix spike duplicates for ammonia, and hardness.

History: CR 06−005: cr. Register April 2008 No. 628, eff. 9−1−08.