Chapter NR 149

LABORATORY CERTIFICATION AND REGISTRATION

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

Note: Certification or registration by the state of Wisconsin under this chapter is not an endorsement or guarantee of the validity of the data generated.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; **correction made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.**

NR 149.02 Applicability. (1) This chapter applies to laboratories applying for certification or registration and laboratories holding valid certification or registration, where department rules require laboratory tests to be done by a certified or registered laboratory.

(2) Section NR 149.21 applies to laboratories applying for certification and laboratories holding valid certifications for the analysis of samples for the safe drinking water program under ch. NR 809.

(3) This chapter does not apply to the certification or registration of laboratories for bacteriological or radiological analyses. Laboratories shall be certified or approved by the department of health and social services for such testing where department rules require the testing to be done by a certified or approved laboratory.

(4) Section NR 149.06 applies to the custodians of the records of any of the following:

(a) A laboratory that currently holds valid certification or registration.

(b) A laboratory whose certification has been revoked, suspended or voluntarily withdrawn.

(c) A laboratory that has not renewed its certification or has transferred ownership.

Note: Administrative Codes and Programs requiring analyses to be done 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, 210– Sewage Treatment Works, 211– General Pretreatment Requirements, 212– Wasteload Allocated Effluent Limits, 219– Analytical Test Methods, 347– Sediment Sampling and Analysis, 508– Landfill Monitoring and Remedial Actions, 605– Identification of Hazardous Waste, 630– Storage, Treatment, and Disposal Facilities, 716– Site Investigation and 809– Safe Drinking Water.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (1) and (2), Register, April, 1988, No. 388, eff. 5–1–88; am. Register, November, 1992, No. 443, eff. 12–1–92; correction in (2) made under s. 13.93 (2m) (b) 7., Stats., Register, April, 1994, No. 460; cr. (4), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.03 Definitions. In this chapter:

(1) "Acceptance limits" means limits established by a reference sample provider which are used to determine if a laboratory has acceptable accuracy.

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

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

(4) "Analyte" means the chemical substance or physical property being tested for in a sample.

(4m) "Analytical staff" means, but is not limited to, the laboratory director, supervisory staff, quality assurance staff, laboratory technicians and chemists.

(5) "Authoritative source" means the following sources:

(a) "Methods for Chemical Analysis of Water and Wastes", EPA–600/4–79–020, Environmental Monitoring and Support Laboratory, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268, Revised 1983, including EPA–600/4–84–017, March, 1984.

(b) "Code of Federal Regulations title 40, Part 136, Appendices A and B", U.S. Government Printing Office, Washington, D.C. 20402, 1987.

(c) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW–846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, November, 1986, including December 1987, July 1992, September 1994 and January 1995 updates.

(d) "Standard Methods for the Examination of Water and Wastewater", 17th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1989.

(e) "1991 Annual Book of ASTM Standards, Section 11.01, 11.02 and 11.04, Water and Environmental Technology", American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

(f) "Procedures for Handling and Chemical Analysis of Sediment and Water Samples", Technical Report EPA/CE–81–1, Environmental Laboratory, U.S. Army Engineer Waterways Experiment Station, P.O. Box 631, Vicksburg, Mississippi 39180.

(g) "Handbook for Sampling and Sample Preservation of Water and Wastewater", EPA–600/4–82–029, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268, September, 1982.

(h) "Techniques of Water–Resources Investigations of the United States Geological Survey, Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", Book 5, Chapter A1, U.S. Geological Survey, Lakewood, Colorado 80225, 1989.

(i) "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA 600/4–79–019, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268, March, 1979.

(j) "Principles of Environmental Analysis", Analytical Chemistry, Volume 55, pages 2210–2218, 1155 16th Street, N.W., Washington, D.C. 20036, 1983.

(k) "Official Methods of Analysis of the Association of Official Analytical Chemists", 15th edition, Association of Official

Analytical Chemists, P.O. Box 540, Washington, D.C. 20044, 1990.

(L) "Methods for the Determination of Organic Compounds in Drinking Water", EPA/600/4–88/039 and EPA/600/4–90/020, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268.

(m) "Methods for the Determination of Metals in Environmental Samples", EPA/600/4–91/010, Office of Research and Development, June 1991.

(n) "Modified GRO– Method for Determining Gasoline Range Organics", WI–PUBL–SW–140, Wisconsin Department of Natural Resources, 101 S. Webster St., Madison, WI, 53707, September 1995.

(o) "Modified DRO– Method for Determining Diesel Range Organics", WI–PUBL–SW–141, Wisconsin Department of Natural Resources, 101 S. Webster St., Madison, WI, 53707, September 1995.

(p) "Quality Assurance Program", USATHAMA PAM 11–41, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, MD 21010–5401, January 1990.

(q) "Technical Notes on Drinking Water Methods", EPA 600/R–94/173, United States Environmental Protection Agency, October 1994.

Note: Copies of these publications are available for inspection at the offices of the department of natural resources, the secretary of state, and the revisor of statutes. Copies of "authoritative sources" listed in pars. (b), (d), (e), (f), (h), (i), (j), and (k) may be obtained at the addresses given. Copies of "authoritative sources" listed in par. (c) may be obtained from the Government Printing Office, Room 190, Federal Building, 517 East Wisconsin Avenue, Milwaukee, Wisconsin 53202. Copies of "authoritative sources" listed in pars. (a), (c), (g), (L), (m) and (q) may be obtained from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487–4650. Copies of "authoritative sources" listed in par. (p) may be obtained from the U.S. Army Environmental Center, Aberdeen Proving Ground, MD, 21020–5401. Copies of "authoritative sources" listed in pars. (n) and (o) may be obtained from the Wisconsin Department of Natural Resources, ERR Section, 101 S. Webster St., Madison, WI, 53707, (608) 261–6424.

(6) "Blind standard" means a standard or a sample with a validated concentration of analyte from an external source in which the concentration of the analyte is unknown to the analyst but is known to the laboratory manager or designee.

(7) "Calibration" means a process used to determine a relationship between the response of the analytical equipment and a known amount of an analyte.

(8) "Certified laboratory" means a laboratory which performs tests for hire in connection with a program which requires data from a certified laboratory, and which receives certification or reciprocal recognition under this chapter.

(8h) "Confirm" means to analyze a sample by a second procedure or with a different chromatography column or detector that verifies the identification of organic compounds.

(8q) "Corrective action" means actions tending or intended to correct a quality control failure.

(9) "Council" means the certification standards review council created under s. 15.107 (11), Stats.

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

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

(13) "Known standard" means a sample prepared or acquired by a laboratory with a known concentration of an analyte used to calibrate or verify the calibration of the analytical system.

(14) "Laboratory" means a facility which performs tests in connection with a program which requires data from a certified or registered laboratory.

Note: A facility consisting of a laboratory and annex within 5 miles of one another may be considered as one laboratory.

(15) "Limit of detection" means the lowest concentration level that can be determined to be statistically different from a blank.

(16) "Limit of quantitation" means the level above which quantitative results may be obtained with a specified degree of confidence.

Note: The limit of quantitation is 10/3 or 3.333 times the limit of detection.

(16m) "MCL" means the maximum permissible level of a contaminant in water which is delivered to any user of a public water system.

(17) "Method of standard addition" means an analytical technique used to quantify samples whose matrices differ significantly from those of the known standards and is accomplished by analyzing the sample and mixtures of the sample with at least 3 known standards, and either plotting the analytical response versus the added concentration and extrapolating the plot to determine the original concentration of the analyte in the sample or by calculating the analytical response for the analyte based upon a least squared regression to determine the original concentration of the analyte in the sample. The samples are processed through all preparative steps prior to the standard additions.

(18) "Method blank" means a sample of reagent grade water which is processed through all preparation steps and the analytical method at the same time and in the same manner as the samples are processed.

Note: When analyzing samples which are other than aqueous matrices the use of a matrix–matched method is advisable.

(19) "On–site evaluation" means an on–site review of the laboratory to determine compliance with this chapter.

(20) "Pesticide" means any of the following:

(a) A pesticide as defined in s. 94.67 (25) and (26), Stats.;

(b) An isomer of a pesticide; or

(c) A degradation product or metabolic product of a pesticide.

(21) "Precision" means the closeness of repeated measurements of the same parameter within a sample.

(21m) "Qualify" means to place a written statement accompanying the test results which identifies anomalies encountered in generating the data.

(22) "Quality control limit" means the calculated acceptance limits determined using a procedure from an authoritative source for replicate and spiked sample analysis or other quality control checks.

(22m) "Raw data" means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of an analysis and are necessary for the reconstruction and evaluation of the analysis which may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, and recorded information from automated collection systems.

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

(24m) "Received on ice" means that sample containers are surrounded by an ice slurry, or crushed, cubed or chipped ice at the time of receipt in the laboratory.

Note: It is acceptable to place the sample containers in plastic bags to preserve sample and label integrity. Alternatives to logging samples as "received on ice" exist in s. NR 149.11 (4).

(25) "Reference sample" means a sample used to determine accuracy prepared by a laboratory other than the laboratory conducting the analysis, in which the true value and acceptance limits are unknown to the laboratory at the time of analysis.

(26) "Registered laboratory" means a laboratory which receives registration or reciprocal recognition under this chapter, does not perform tests commercially for hire and which:

(a) Performs tests in connection with a program which requires data from a registered laboratory; and

(b) Performs tests solely on its own behalf or on behalf of a subsidiary or other corporation under common ownership or control, or is owned or controlled by a municipality or 2 or more munici-

palities and performs tests solely on behalf of the municipality or municipalities.

(26g) "Replicate sample" means 2 equal aliquots taken from the same sample container and analyzed independently for the same constituent.

(26r) "Revocation" means cancellation of a laboratory's certification or registration.

(27) "Results" includes measurements, determinations and information obtained or derived from tests.

(28) "Sample matrix" means the general physical–chemical makeup of the sample.

Note: Wastewater samples, water supply samples, waste samples, surface water samples, groundwater samples, sediment samples, and soil samples may have different physical–chemical makeups.

(28m) "Sensitivity" means the ability of a method or instrument to detect an analyte at a specified concentration.

(29) "Matrix spike" means a replicate sample to which a known amount of the analyte has been added prior to any preparative steps to determine percent recovery.

(29m) "Suspension" means a temporary cancellation of a laboratory's certification which does not require an on–site evaluation for reinstatement.

(30) "Test" means any chemical, bacteriological, biological, physical, radiation, or microscopic test, examination or analysis conducted by a laboratory on water, wastewater, waste material, soil or hazardous substance.

(31) "Test category" means one type of test or group of tests specified under s. NR 149.04 for similar materials or classes of materials, or which utilize similar methods or related methods.

(31m) "Temperature blank" means a sample container of at least 40 ml in volume which is filled with water and transported along with each batch of samples in order to determine the temperature of the samples at the time of receipt at the laboratory.

(32) "Trip blank" means a sample of reagent grade water or methanol which is used to determine possible contamination of sample bottles from volatile organic chemicals while in transit to and from the laboratory.

(33) "Unfamiliar sample" means a sample for which the laboratory has either no information or questionable information from previous characterizations of samples from the same source, or a sample for which there is no information on the process generating it.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; r. and recr. (5) (b), Register, August, 1989, No. 404, eff. 9–1–89; am. (5) (a) to (c), (g) to (i), (k), (6), (13) to (17), (22) and (29), r. and recr. (5) (L) and (18), cr. (5m), (8h), (8q), (21m), (22m), (26g), (26r), (29m) and (33), r. (11), Register, November, 1992, No. 443, eff. 12–1–92; r. (23), eff. 1–1–93; cr. (4m), (16m), am. (5) (c), Register, April, 1994, No. 460, eff. 5–1–94; am. (15) and (16), Register, August, 1995, No. 476, eff. 9–1–95; am. (5) (c), (17), (29) and (32), cr. (5) (n) to (q), (24m), (28m) and (31m), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.04 Test categories. (1) Test categories are contained in Table 1. Listed with each test category are the specific analytical test analytes included in that test category and the key analyte which is the analyte which will be required for the reference sample analysis. A laboratory may apply for certification or registration in any or all of the test categories. If an analyte is listed in more than one test category, the laboratory may apply for certification or registration in any of the test categories including that analyte.

(2) The safe drinking water test category has specific requirements which are described in s. NR 149.21.

(3) The effluent toxicity test category has specific requirements which are described in s. NR 149.22.

TABLE 1

Test Categories

No.	Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
1.	Oxygen Utilization	Total BOD_5	Biochemical oxygen demand, carbonaceous biochemical oxygen demand.
2.	Nitrogen	Each analyte for which certification or registration is desired except nitrite.	Nitrate as Nitrogen, Nitrite as Nitrogen, Ammonia as Nitrogen, total Kjeldahl Nitrogen, Nitrate +Nitrate as Nitrogen.
3.	Phosphorus	Total Phosphorus	Orthophosphate, Phosphorus.
4.	Physical	Total Suspended Solids	Total Solids, Dissolved Solids, Volatile Solids, Total Suspended Solids, Oil and Grease.
5.	General I	Hardness	Alkalinity/Acidity, Bromide, Chlorophyll a, Color, Hardness, Silica, Silicate, Sulfide, Sulfite,Surfactants.
6.	General II	Each analyte for which certification or registration is desired.	Chemical Oxygen Demand, Chloride, Cyanide, Fluoride, Sulfate, Total Phenolic Compounds.
7.	General III	No reference sample	Extraction Procedure (EP) Toxicity, Ignitability, Reactivity, Total Releasable Cyanide, Total Releasable Sulfide,Corrosivity, Waste Fingerprinting Analyses, Total Organic Carbon, Total Organic Halide, Toxicity Characteristic, Leaching Procedure (TCLP), Synthetic Precipitation Leaching Procedure (SPLP). Note: Certification or registration for EP, TCLP, or SPLP under this test category is only for the extraction and does not include the analytes.

TABLE 1
Test Categories

No.	Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
8.	Metals I	Each analyte for which certification or registration is desired.	Aluminum, Antimony, Arsenic, Barium,Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, and Zinc.
9.	Metals II	Each analyte for which certification or registration is desired.	Bismuth, Gold, Iridium, Lithium, Osmium, Palladium, Platinum, Rhodium, Ruthenium, Silicon, Titanium, Tungsten, and Zirconium.
10.	Organics; Purgeable by Gas Chromatography or Gas Chromatography/Mass Spectrometry	Representative purgeable analytes.	Purgeable Halocarbons, Purgeable Aromatics, Acrolein, Acrylonitrile.
11.	Organics; Semivolatiles by Gas Chromatography	Representative analytes within each subcategory for which certification is desired. The following subcategories are included: Phenolic Compounds (acid–extractables), Phthalate Esters, Nitrosamines, Nitroaromatics and Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons.	Phenolic Compounds (acid–extractables), Phthalate Esters, Nitrosamines, Nitroaromatics and Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons.
12.	Organics; Semivolatiles by Gas Chromatography/Mass Spectrometry	Representative analytes within each subcategory for which certification is desired including Phenolic Compounds. (Note: All semivolatiles included in a particular study must be analyzed and reported. To be considered a representative sample for base/neutral extractables, a study must include at least 4 subcategories of base/neutral extractables.)	Phenolic Compounds (acid–extractables) and Base/Neutral Extractable Compounds (excluding pesticides).
13.	Organics; Extractables by Liquid Chromatography	Representative Polynuclear Aromatic Hydrocarbons, Benzidines, and Pesticides analyzable by liquid chromatography.	Benzidines, Polynuclear Aromatic Hydrocarbons, Aldehydes and Ketones, Carbamate Pesticides subject to Liquid Chromatography (e.g., carbofuran, oxamyl, and methomyl) and Other Pesticides subject to Liquid Chromatography (e.g., diquat and paraquat).
14.	Organics; Pesticides	Representative pesticides within each subcategory for which certification is desired.	Acid Herbicides (e.g., 2,4–D,2,4,5–T, picloram, etc.), Nitrogen Pesticides, Organophosphorus Pesticides, Triazine Pesticides (including metabolites) and Other Pesticides.
15.	Organics; Petroleum Hydrocarbons	Gasoline Range Organics (GRO), Diesel Range Organics (DRO), Petroleum Volatile Organic Compounds (PVOC).	Gasoline Range Organics, Diesel Range Organics, Petroleum Volatile Organic Compounds (PVOC).
16.	Organics; Organochlorine Compounds	Representative analytes within the Aroclors and Organochlorine pesticides groups for which certification or registration is desired.	Polychlorinated Biphenyls and Organochlorine Pesticides.
17.	Organics: Polychlorinated Dibenzo–P–Dioxin	No reference sample; for each analyte for which certification or registration is desired the accuracy and precision data (acceptable according to an authoritative source) shall be submitted to demonstrate the ability to perform the analysis. See s. NR 149.13 (11).	Polychlorinated Dibenzo–P–Dioxin, Polychlorinated Dibenzo–P–Furan.
18.	Safe Drinking Water	Each analyte or analyte group for which certification is desired.	Cyanide, Fluoride, Metals, Nitrate as Nitrogen, Nitrite as Nitrogen, Nitrate Nitrite as Nitrogen, Synthetic Organic Contaminants, Total Trihalomethanes, Volatile Organics. Note: The list of certifiable parameters within each group is given on the application form.

TABLE 1
Test Categories

No.	Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
19.	Any Single Analyte or Group of Analytes	That Analyte or Analytes from that Group (where reference samples are available).	Per Request.
20.	Effluent Toxicity	No reference sample	Acute Invertebrate Toxicity, Acute Vertebrate Toxicity, Chronic Invertebrate Toxicity, Chronic Vertebrate Toxicity.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. No. 404, Register, August, 1989, No. 404, eff. 9–1–89; am. (2), Register, November, 1992, No. 443, eff. 12–1–92; am. table 1, eff. 1–1–93; cr. (3), eff. 7–1–93; am. Table 1, items 7, 15, 18, Register, April, 1994, No. 460, eff. 5–1–94; am. Table 1 items 2, 10 and 13, r. and recr. items 11, 12 and 14, Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.05 Fees. (1) ANNUAL FEES. (a) An annual fee shall be assessed to each laboratory holding a certificate for certification or registration. The department shall set a schedule of fees for certified and registered laboratories which are designed to recover the costs of administering this chapter.

(b) The total fee income shall be designed to generate revenues equal to the department of administration's approved spending authority for this program. The department may adjust the fee schedule according to the formulas in subds. 1. to 4. and the relative value items in table 2. Annual fee adjustments shall be reviewed by the laboratory certification standards review council and approved annually by the natural resources board.

1. Fee Income $\leq$ ASA – TR

a. Fee income is the total of all fees (including renewals, applications, reciprocity, and late fees) that are collected in a given fiscal year.

b. TR is the total out–of–state travel reimbursements 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 par. (c).

Note: The department of administration approved spending authority is given in s. 20.370 (2) (fj), Stats., and may be revised by the department of administration to cover actual program cost.

2. Total # RV Units = $\sum$ (# Labs 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 of each fee item (RV of item) is listed in table 2.

b. # Labs in item is a count of how many labs 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 (# labs in item) for each individual 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 of item) (Cost per RV)

Note: If the cost per RV is $25, an item with an RV of 10 would cost $250.

(c) The fees for the upcoming fiscal year shall be set 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 one month 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.

(d) The minimum annual certification fee applies to laboratories certified in any of the test categories 5 through 19, except for laboratories certified only for nitrate + nitrite in test category 18, for which there is no minimum annual fee. There is no minimum fee for registration. The department may adjust this fee by the procedures given in pars. (a) to (c).

Table 2
Fees for Certification and Registration

Item	Relative Value[1]
1. Base Fee	10
2. Cat. 1 – Oxygen Utilization	1
3. Cat. 2 – Nitrogen	1
4. Cat. 3 – Phosphorus	1
5. Cat. 4 – Physical	1
6. Cat. 5 – General I	2
7. Cat. 6 – General II	2
8. Cat. 7 – General III	4
9. Cat. 8 – Metals I	4
10. Cat. 9 – Metals II	4
11. Cat. 10 – Purgeable Organics	4
12. Cat. 11 – Semivolatiles by GC	4
13. Cat. 12 – Semivolatiles by GC/MS	4
14. Cat. 13 – Liquid Chromatography	4
15. Cat. 14 – Pesticides	4
16. Cat. 15 – Petroleum Hydrocarbons	12
17. Cat. 16 – Organochlorine Compounds	4
18. Cat. 17 – Dioxins	12
19. Cat. 18 – Safe Drinking Water	20
20. Cat. 18– NO_3+NO_2 only	2
21. Cat. 19 – Any Single Analyte	4
22. Cat. 20 – Effluent Toxicity Testing	26
23. Initial Application	6
24. Revised Application	3
25. Minimum Annual Certification & Reciprocity[2] Fee	24
26. Late Renewal Fee (assessed 30 days after payment due date)	2
27. Evaluation of Out–of–State Labs	Additional Travel Costs
28. Enforcement Follow–up Evaluation	Actual Cost of Evaluation
29. Discretionary Acceptance	Actual Cost of Determining Data Quality

[1] The relative value (RV) of each item was calculated based upon the fee schedule from fiscal year 1995, where one RV equaled $25.

[2] Upon initial application for reciprocity the laboratory shall pay the reciprocity fee and the initial application fee.

(2) REFUNDS. Fees are not refundable, except for overpayment.

(3) USE OF FEES. Fees shall be used to offset the cost to the department for certification and registration of laboratories, laboratory evaluations, discretionary acceptance of data, reciprocity, and collection of fees.

(4) FEE REVISION. The department may amend the formulas in this section based upon a demonstrated need for revision to support the level of effort in the program. Any amendments to the formulas in this section shall be reviewed by the laboratory certification standards review council prior to being proposed as rule amendments.

Note: The approved spending authority reflects the amount of money that the program may spend in a given fiscal year and is set by the legislature as part of the biennial budget process. The department of administration may adjust this amount to reflect actual program costs, such as increases to pay plans, benefits or additional positions approved by the legislature.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (1) (b) and (c), (2), (3) and (5), r. and recr. (1) (g), Register, November, 1992, No. 443, eff. 12–1–92; cr. (1) (h) and (i), eff. 1–1–93; r. and recr. (1), Register, April, 1994, No. 460, eff. 5–1–94; r. and recr. (1), (4) and Table 2, r. (5), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.06 Records. (1) Certified and registered laboratories or their trustee shall retain records generated during a certification period for 3 years following the date of analysis. The records shall be available for review upon request of the department. The department may require by written notice that this period be extended if the department has initiated legal action involving the test results. Records to be retained include but are not limited to records of the following:

(a) Samples processed so that any sample may be traced back to the analyst, date collected, date analyzed, and method used including raw data, intermediate calculations, results, and the final report.

(b) Quality control date for spikes, replicates, method blanks, blind standards, reference samples, calibration standards and known standards. Quality control results shall be traceable to all of the associated sample results.

(c) Quality control limits for spikes and replicates.

(d) Information on maintenance of laboratory instruments.

(e) Preservation status of samples on arrival.

(f) Corrective actions as required in s. NR 149.14 (3) (k).

(g) Log books, bench sheets, journals or notes necessary to demonstrate that method or legal requirements have been met.

(2) The following records shall be retained by the person doing the sampling for a period of 3 years from the date of analysis. The department may require by written notice that this period be extended if the department has initiated legal action involving the test results.

(a) Sample preservation procedures if different than specified by the methodology.

(b) The following general sampling information:

1. Whether the sample was a grab sample or composite sample for wastewater samples.

2. If the sample was a composite wastewater sample, whether it was flow or time proportional.

3. Whether the sample was filtered in the field for groundwater monitoring well samples.

4. Any unusual circumstances that may affect the sample results.

5. Results of field analyses, if done.

6. Location, date, collector's name and time of sampling.

(3) The laboratory and the person doing the sampling shall submit copies of records required to be retained under subs. (1) and (2), respectively, upon request of the department.

(4) Upon the department's request, a certified or registered laboratory shall submit to the department records, under sub. (1), from any subcontracted laboratories.

(5) Records described under subs. (1) and (2) shall be handled in a manner to ensure their permanence and security. Handwritten records shall be recorded in ink. Electronic records may be allowed if the process safeguards against corruption, loss and inappropriate alterations.

Note: Chapter NR 809, safe drinking water program requires that the actual chemical sampling results be retained for 10 years by the agency responsible for the drinking water supply.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (1) (intro.) to (c), r. and recr. (4), Register, November, 1992, No. 443, eff. 12–1–92; am. (2) (b) 6., cr. (1) (e) to (g) and (5), eff. 1–1–93; am. (1) (intro.), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.07 Application for certification or registration. (1) APPLICATION. In order for a laboratory to apply to become certified or registered, the laboratory shall:

(a) Complete an application and submit it with the appropriate fees prescribed in s. NR 149.05. Incomplete applications and applications received without the appropriate fees may be returned to the applicant unprocessed if any of the information required in this subsection is not included with the application.

Note: Application forms are available from the Department of Natural Resources, Bureau of Integrated Science Services, P.O. Box 7921, Madison, WI 53707.

(b) Specify the test categories for which certification or registration is desired. Once a laboratory is certified or registered, if the laboratory wishes to become certified or registered in additional test categories, the laboratory shall submit to the department:

1. The test category for which certification or registration is requested;

2. The test category fee for each additional test category;

3. Acceptable reference sample results when required under s. NR 149.13.

(c) Specify the methodology to be used to analyze for each test anticipated to be processed by the laboratory within each test category for which certification or registration is requested. This methodology shall be acceptable under s. NR 149.11.

(d) Agree to comply with this chapter.

(e) Agree to allow the department or its representative to inspect the laboratory to determine compliance with this chapter, with prior notice except as provided in s. NR 149.41 (1).

(f) Submit to the department acceptable results on reference samples for test categories requiring reference samples. The laboratory shall provide acceptable results on 2 consecutive reference samples if unacceptable results are obtained on 3 consecutive reference samples for the same analyte or analyte group.

(g) Submit other analyte specific information as required by the method or the department.

Note: Other analyte specific information may include detection limit studies and initial demonstrations of laboratory capability where required by the analytical methods.

(2) EXPIRATION OF APPLICATION. If the laboratory has not submitted all the necessary materials described in sub. (3) (a) to (c), its application shall expire one year from the date the application is received by the department. If the laboratory then wishes to pursue certification or registration, the laboratory shall pay the appropriate fees and submit a new application per this section.

(3) APPLICATION REJECTION. A laboratory may not apply and the department may not accept application for additional certifications or registrations or reapplications when:

(a) A notice of violation has been issued for violations of this chapter, and the problems causing enforcement have not been corrected.

(b) An administrative order has been issued for violations of this chapter, the problems causing enforcement action have not been corrected and the time period of suspension or revocation is in effect.

(c) A laboratory is not in compliance with this chapter at the time it voluntarily relinquishes its certification or registration, problems existing prior to relinquishing its certification or registration have not been corrected and 6 months have not elapsed since the voluntary action was undertaken.

(4) EVALUATION. For a laboratory to become certified or registered, successful completion of an on–site laboratory evaluation is required. The on–site evaluation of an applicant laboratory shall be completed within 90 days from receipt of materials specified under sub. (3) (a), (b) and (c) unless mutually agreed upon by the applicant laboratory and the department. Once a laboratory is certified or registered, if the laboratory wishes to become certified or registered in additional test categories, the department may waive the requirement for an on–site laboratory evaluation.

(5) ISSUANCE OF CERTIFICATION OR REGISTRATION. The department shall issue the certification or registration to the applicant within 20 business days of receipt of the completed application. The application is not considered to be complete until all of the following requirements are satisfied:

(a) Receipt of the completed application form as described in subs. (1) and (2),

(b) Payment of the annual fee,

(c) Successful performance on reference samples, and

(d) Successful completion of an on-site evaluation.

(6) RENEWAL OF CERTIFICATION OR REGISTRATION. (a) Certifications and registrations shall be renewed prior to July 1 of each year. If the laboratory uses the discharge monitoring report quality assurance samples for any or all of its reference samples, then the renewal date shall be prior to January 1. Prior to the renewal date the department shall, by letter, request each certified or registered laboratory to submit the fee for the next year, reference sample results, and to indicate changes in the laboratory's certification or registration status.

(b) In order to renew certification or registration, the required fee shall be paid and the laboratory shall have acceptable reference sample results prior to renewal.

(c) Certification or registration shall be expired for laboratories not meeting the criteria given in par. (b) within 60 days after the payment due date or at the certification expiration date for laboratories expiring in December of the fiscal year.

(7) APPLICATION FOR TRANSFER OF LABORATORY CERTIFICATION OR REGISTRATION. (a) This subsection applies to a change in ownership that involves the purchase or lease of equipment and where greater than or equal to 60% of the analytical staff are retained. A change in ownership that involves the purchase or lease of equipment and where less than 60% of the analytical staff are retained shall be treated as an application under subs. (1) to (3).

(b) Within 10 days following a change in laboratory ownership, the new owner of a certified or registered laboratory shall notify the department in writing about the change. Within 40 days following the change in laboratory ownership, a certified or registered laboratory shall do all of the following:

1. Submit a revised application, indicating any changes in the equipment, methodology and staffing.

2. Pay the revised application fee.

3. Agree to allow the department or its representative to inspect the laboratory to determine compliance with this chapter, with prior notice except as provided in s. NR 149.41 (1).

4. Agree to comply with this chapter.

5. In the event that the laboratory has not resolved deficiencies identified by the department or if there is an outstanding enforcement action against the laboratory, the new owner shall agree to correct conditions which led to the deficiencies or enforcement action in accordance with a schedule which is acceptable to the department.

(c) All open or pending enforcement actions shall be transferred with the certification or registration.

(d) Failure to meet the conditions specified in par. (a) shall cause the previous certification or registration to expire.

(e) The laboratory may operate under the previous certification or registration until the department notifies the laboratory on the acceptance or rejection of the transfer application. If the department rejects the transfer application, the laboratory is no longer certified or registered and the new owner shall submit an application under subs. (1) to (3). The department shall conclude the review of the transfer application within 20 business days of the receipt of the completed transfer application.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; cr. (1m), r. (5), Register, November, 1992, No. 443, eff. 12–1–92; am. (1) (b) 3., (c), (e), (f) and (4), eff. 1–1–93, r. and recr. (2) and (3), eff. 7–1–93; cr. (1g), (5), Register, April, 1994, No. 460, eff. 5–1–94; am. (1) (a), renum. (1g) to (5) to be (2) to (7), cr. (1) (g) and (6) (c), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.11 Methodology for laboratory analysis, sample collection, sample preservation, and holding time. (1) The analytical methodology used for a specific test shall:

(a) Be appropriate for the test and sample matrix.

(b) Be the analytical methodology required by applicable state and federal regulations.

(c) Be selected from an authoritative source specified by the department if methodology is not prescribed by state and federal regulations. When methods are not available in authoritative sources that meet the needs of the department, the department may specify or allow methods from other sources.

(d) Enable the laboratory to quantitate at levels required by the department. If the required level cannot be met by the methods available under par. (b) or (c), then the method with the lowest limits of detection shall be selected.

(e) Be available to the analyst.

Note: Analytical methodologies required by state rules are in chs. NR 219, 508, 605, 675, 700 and 809. Those required by federal regulations are in 40 CFR 136, 141, and 268.

(2) Sample collection methods required by applicable state and federal law shall be followed. If the sampling method for the test is not specified by state or federal law, it is recommended that authoritative sources be followed for sampling procedures.

Note: Sample collection methods required by state rules are in chs. NR 218 and 140.

(3) Sample preservation procedures and holding times required by state and federal regulations shall be followed. If the sample preservation procedures and holding times are not required by state or federal regulations, the sample preservation procedures and holding times established in the analytical methodology shall be followed. If the analytical methodology does not establish sample preservation procedures or holding times, procedures in the authoritative sources shall be followed.

Note: Sample preservation procedures and holding times are given in 40 CFR 136, ch. NR 219 "Test Methods for Evaluating Solid Waste" as cited in s. NR 149.03 (5) (c), and may be specified in the analytical methods.

(4) Samples requiring preservation at 4°C under this section may be recorded as "received on ice" only if solid ice is present in the cooler at the time the samples are received. Samples cooled during shipping with ice packs may not be recorded as received on ice. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

(a) The temperature of an actual sample.

(b) The temperature of a temperature blank shipped with the samples.

(c) The temperature of the melt water in the shipping container.

(5) The limit of quantitation and limit of detection shall be determined for each analyte reported by a laboratory in accordance with a method specified by the department. The department may also require that limit of detection be determined for a specific matrix.

(6) When a method of analysis specifies a validation procedure, the validation procedure shall be completed before samples can be analyzed and reported to the department. The results of this validation procedure shall be documented and kept on file for 3 years.

(7) A copy of the methodology used by the laboratory for each analyte analyzed shall be available to the analyst.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (5), Register, August, 1989, No. 404, eff. 9–1–89; r. and recr. (1), am. (3) and (5), r. (4), cr. (6) and (7), November, 1992, eff. 12–1–92; am. (5), Register, August, 1995, No. 476, eff. 9–1–95; am. (3), cr. (4), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.12 Alternate methodology. (1) EPA APPROVAL. The department may permit the use of alternate methodologies other than those prescribed in this chapter if EPA has granted an approval for their use. The laboratory shall submit to the department a copy of EPA's written approval for the use of the alternate method.

Note: Alternate methodology approval by EPA is required by ch. NR 219 and by federal regulations in 40 CFR 136, 141, 260 and 403.

(2) EMERGING TECHNOLOGY. The department may allow alternate methods which use existing new or innovative technologies on a case–by–case basis. Laboratories may request approval for an emerging technology method by following the 2 step approval process outlined in pars. (a) and (b). Modifications to an approved method may not be considered an emerging technology. Laboratories shall request approval for modifications to an approved method according to sub. (1).

(a) Initial requests for using emerging technology methods shall be made to the laboratory certification program. The request shall include the reasons for proposing the method and the potential scope of use for the method.

(b) The department may approve or deny the request within 90 days based on a demonstrated department need for the emerging technology method. If the request is granted, the department will establish criteria for validating the method on a case–by–case basis. If the method validation meets the predetermined criteria, the department shall permit the use of the method. The department may charge a fee under s. 299.11 (5) (d), Stats., if it is necessary to verify the results of the data.

Note: Emerging technology as defined requires that the method use principles of sample preparation, detection or quantitation that are not found in an approved method. If the department grants an approval to develop the method, the criteria for its use and the scope of its use will be defined in a method development summary. Most alternate methodologies proposed as emerging technologies will only be approved for use on a particular project or type of project, such as field work.

History: Cr. Register, April, 1986 No. 364, eff. 5–1–86; r. and recr. Register, November, 1992, No. 443, eff. 12–1–92; renum. to be (1) and am., cr. (2), Register, February, 1996, No. 482, eff. 3–1–96; **correction in (2) (b) made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.**

NR 149.13 Reference samples. (1) Laboratories applying for certification or registration shall analyze reference samples where required for each test category for which the laboratory applies for certification or registration. In order to become certified or registered the reference sample results shall meet the acceptance limits calculated by the reference sample provider. The reference sample acceptance limits of the provider and the units of concentration shall be provided to the department with the reference sample results.

(2) Where certification or registration in a test category is based on more than one analyte, the laboratory shall have at least 80% of the results acceptable to be certified or registered for the test category.

(3) A certified laboratory shall successfully analyze and report results of one reference sample for each test category for which the laboratory seeks certification. The department may require a maximum of 3 reference samples per year for each of the test categories for which the laboratory seeks certification. A registered laboratory shall successfully analyze and report results of one reference sample per year for each test category for which the laboratory seeks registration. Reference samples shall be obtained from the Wisconsin state laboratory of hygiene or a source approved by the department. The department shall maintain a list of approved reference samples sources. Criteria for approving providers include all of the following:

(a) The means of calculating the acceptance limits shall be at least as stringent as those used by the Wisconsin state laboratory of hygiene.

(b) The acceptance limits are provided to the laboratory by the reference sample provider after the sample results and acceptance limits are provided to the department.

(c) The reference sample provider agrees that the acceptance limits or the true value will not be provided to the laboratory before it is provided to the department.

(4) For renewal of certification or registration, reference samples from an approved source shall be analyzed and reported to the reference sample provider. If the results of this reference sample do not meet the acceptance limits, analysis of an additional reference sample may be required under sub. (7).

(5) A laboratory's results are acceptable if they are within the reference sample provider's acceptance limits.

(6) The Wisconsin state laboratory of hygiene shall use standard statistical methods, with the concurrence of the council, to determine the acceptance limits.

(7) If a laboratory does not meet the acceptance limits of the reference sample provider, the department may investigate the reason for the failure and require a second reference sample. The laboratory shall analyze and report the results for the second sample to the department within 30 days of receipt of the second sample, unless an extension is requested and granted. If the second reference sample results do not meet the acceptance limits, the department may initiate an assessment of the laboratory's quality control records.

(8) Within 30 days of the date of notification of the second failure to meet acceptance limits on a reference sample, the laboratory shall initiate, with the department's approval, an action plan to correct the problems. This action plan shall include a timetable for correcting the problems.

(9) After the laboratory takes corrective action, it shall analyze a third reference sample within the timetable approved by the department.

(10) Registered laboratories shall qualify the tests results of the analytes in the test categories in which the laboratory has failed to meet acceptance limits on 2 consecutive reference samples. Certified laboratories may be required to qualify the test results of the analytes in the test categories in which the laboratory has failed to meet acceptance limits on 2 consecutive reference samples.

(11) For test category 17, no reference sample is required. The laboratory shall demonstrate, upon application for certification or registration, acceptable precision and percent recovery based on the successful completion of the initial precision and accuracy portion of the method. The department shall judge acceptability based on the criteria given in the method.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (1), r. and recr. (6), Register, November, 1992, No. 443, eff. 12–1–92; am. (3) to (5), (7), (10), (11) (intro.) and (6), r. and recr. (2), eff. 1–1–93; am. (11), Register, April, 1994, No. 460, eff. 5–1–94.

NR 149.14 Quality control. (1) Each laboratory shall maintain a quality control program. The quality control program shall include a written quality assurance plan. The quality control data shall be documented and such documents shall be available, upon request, to the department.

(3) At a minimum, the quality control program shall consist of:

(a) Calibration and maintenance of all test instruments and equipment as necessary to maintain accuracy.

(b) A calibration done or a known standard analyzed on each analysis day. The instrument response for the known standard shall be within the pre–established limits under par. (c). A calibration shall consist of at least 3 standards and a blank except as allowed in approved methods using ion selective electrodes or inductively coupled plasma.

Note: Using only 3 calibration standards presumes that the working range is within a limited linear region of the curve for the analyte of concern. The actual number of calibration points used should be based upon the width of working range and the shape of the calibration curve and should insure the accuracy of the determination. For most inorganic analyses, the blank is included in the calibration curve. A correlation coefficient of at least 0.995 generally indicates acceptable characterization of the curve; however, for some organic analyses a correlation coefficient of at least 0.990 can be more reasonably expected. For analyses requiring a higher degree of accuracy, additional standards and a higher correlation coefficient are desirable.

(c) A known standard analyzed after the analysis of 20 samples, if 20 or more samples are analyzed in an analysis day. The instrument response for the known standard shall be within the following pre–established limits:

1. For test categories 2, 3, 6, 8, 9 and for total organic carbon, total organic halide, and hardness, the pre–established limit shall be ± 10%, unless an approved method specifies otherwise.

2. For test categories 10, 11, 12, 13, 14, 15, 16, and 17, the pre–established limits shall be ± 15%, unless an approved method specifies otherwise.

3. There is no requirement to analyze a known standard for alkalinity/acidity, color, odor and analysis under test category 4.

4. For test category 1, a known standard shall be analyzed after the analysis of 20 samples or once a week. The known standard for biochemical oxygen demand shall be glucose/glutamic acid. The limits on this quality control check shall be as established in an authoritative source or those established by the provider, whichever is more stringent.

5. For test category 19 the pre–established limit shall be appropriate for the test.

(d) At least one method blank shall be prepared or analyzed, or both on each analysis day, for those tests for which method blanks are appropriate. Certain methods require that a nonreacted sample be used as a blank. Method blanks may not be used to correct sample results, except when specified in the method. There is no requirement to run a blank for solids testing performed under test category 4. The method blank results exceed control limits when results are higher than the highest of any of the following.

1. The limit of detection.

2. Five percent of the regulatory limit for that analyte.

3. Five percent of the measured concentration in the sample.

(e) A replicate sample shall be run after the analysis of 20 samples for each matrix type, unless the methodology specifies otherwise. No replicate samples are needed for oil and grease.

(f) Spiked samples shall be analyzed for each matrix type. The samples shall be spiked before any extraction or digestion. The frequency of spiked analysis shall be as cited in the approved method or authoritative source. If no frequency is given, then the minimum frequency shall be:

1. After the analysis of 10 samples, for test categories 10 to 17, 19, total organic halide and total organic carbon.

2. After the analysis of 20 samples, for test categories 2, 3, 5, 6, 8, and 9.

3. No spiked analysis is required for test categories 1 and 4 and for alkalinity/acidity, chlorophyll a, color, sulfide, sulfite, ignitability, reactivity, and gravimetric tests, or tests where appropriate standards are not available for spiking.

4. Samples for analysis by the toxicity characteristic leaching procedure (TCLP) or EP toxicity must be spiked after the extraction at the frequency cited in this paragraph.

(g) Quality control limits for replicate sample and spiked sample analysis shall be calculated for each matrix type using a method from an authoritative source. When quality control data shows a dependency on concentration, the laboratory shall calculate separate control limits to address the concentration dependency. For laboratories with less than 20 quality control results within 12 months, the laboratory may set quality control limits based on information given in the authoritative sources, laboratory experience, or the experience of other laboratories.

(h) If the results of known standards, spiked samples, method blanks or replicates exceed the quality control limits, corrective action shall be taken by the laboratory. The laboratory shall reanalyze the affected samples or qualify the results back to the last acceptable quality control check of the same type unless the laboratory determines that sample results are unaffected. The results are qualified by reporting that the laboratory analysis was not within the acceptance limits for this test.

(i) If the analysis of a spiked sample exceeds the quality control limits, corrective action shall be taken by the laboratory. If it is determined by the laboratory that the discrepancy has affected past sample results, the laboratory shall reanalyze the samples or qualify the results, for those samples of the same sample matrix, back to the last acceptable quality control check. The results are qualified by reporting that the laboratory analysis was not within acceptance limits for this test. The impact of the spiked sample results on samples of different sample matrices shall be examined to insure that whatever affected the spiked sample had no impact on those samples of different matrices.

(j) Blind standards shall be analyzed 3 times a year if a standard is available and the analyte was analyzed during the previous 4–month period. Analysis of blind standards shall meet all of the following requirements:

1. Analysis shall be conducted for each analyte in test categories 1 to 9, 15, and 19.

2. Analysis shall be conducted for one analyte in each test category in test categories 10 to 14, 16, and 17.

3. If the result for any analyte does not fall within the limits established by the provider or the laboratory, corrective action shall be taken by the laboratory and an additional blind standard shall be analyzed to verify that the corrective action was successful.

4. The blind standard shall be analyzed at least 3 months and no longer than 5 months after the previous blind standard.

(k) Where replicate, spikes, and other quality control limits are exceeded, documentation shall be available to the department, upon request, indicating what corrective action was taken to bring the results back within limits.

(5) If it has been determined that an organic analyte is present in an unfamiliar sample, the laboratory shall confirm the results, unless the analysis is done by mass spectrometry.

(6) The quality control requirements of subs. (3) and (5) do not apply to non–trace level analyses conducted under a waste analysis plan required by s. NR 630.13 for treatment, storage and disposal facilities or for other facilities required to prepare a waste analysis plan in accordance with the requirements specified in s. NR 630.13. At a minimum, the quality control specified by the methodology cited in the approved waste analysis plan shall be followed.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (3) (c) 4., Register, April, 1988, No. 388, eff. 5–1–88; r. (2) and (4), cr. (3) (f) 4., (j) 4. and (6), Register, November, 1992, No. 443, eff. 12–1–92; am. (1), (3) (a) to (f) 3., (g), (h), (j), (k) and (5), r. (3) (i), eff. 1–1–93; am. (3) (d) and (h), Register, August, 1995, No. 476, eff. 9–1–95; am. (3) (c) 4., (e) and (f) (intro.), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.15 Data reporting. With each set of sample results, a laboratory shall report:

(1) The condition and temperature of improperly preserved samples upon receipt in the laboratory. If the holding time of the sample exceeds the maximum holding time required under this chapter, the laboratory shall report this fact with the results.

(2) The identities of all laboratories, if any, subcontracted to perform analyses for the sample set.

(3) All analytical results greater than the limit of detection, as determined by a method specified by the department. All analytical results greater than the limit of detection and below the limit of quantitation shall be appropriately qualified.

Note: The requirement in sub. (3) becomes effective January 1, 1997 only for those substances with standards specified in chs. NR 105, 140 and 720 that are below the applicable limits of quantitation. Chapter NR 809 requires that this information be reported for all regulated primary drinking water contaminants. The department shall annually publish a list of these substances. Laboratories shall use the best available analytical science to determine whether, in their best professional judgment, a substance has been detected.

History: Cr. Register, February, 1996, No. 482, eff. 3–1–96, except (3) eff. 1–1–97.

NR 149.21 Requirements for safe drinking water certification. This section applies to those laboratories certified under test category 18 and is for the purpose of qualifying laboratories to perform compliance monitoring under ch. NR 809.

(1) GENERAL REQUIREMENTS FOR SAFE DRINKING WATER CERTIFICATION. (a) The criteria and procedures for safe drinking water certification are those criteria and procedures specified in "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA/570/9–90/008, third edition, EPA, Office of Water, April 1990, including change 1, October 1991 and change 2, September 1992.

(b) The laboratory shall determine limits of detection according to the procedures in 40 CFR 136 Appendix B.

(c) The laboratory shall meet the criteria specified in the initial demonstration of capability from the approved method of analysis.

(2) FLUORIDE. Fluoride analyses required under s. NR 809.705 need not be performed by a certified laboratory.

(3) FREE CHLORINE RESIDUAL. Free chlorine residual and total chlorine residual analyses required under s. NR 809.705 need not be done by a certified laboratory.

(4) ANALYSIS FOR PH. Analyses for pH required under s. NR 809.14 need not be done by a certified laboratory.

(5) TURBIDITY. Turbidity analyses as required under s. NR 809.41 need not be done by a certified laboratory.

Note: 40 CFR 141.28 excludes turbidity, free chlorine residual and pH from certification.

(6) REQUIREMENTS FOR INORGANIC CHEMICALS. To receive certification to conduct analyses for cyanide, fluoride, metals, nitrate, and nitrite the laboratory shall:

(a) Analyze reference samples for these substances, provided by EPA, and achieve quantitative results on the analyses that meet the acceptance limits listed in 40 CFR 141.23 (k) (6) (ii) and 141.89 (a) (1) (ii).

(b) Achieve acceptable limits of detection as specified in 40 CFR 141.23 (a) (4) (i) and 40 CFR 141.89 (a) (1) (iii) or 10% of the MCL, whichever is greater.

(7) REQUIREMENTS FOR VOLATILE ORGANIC COMPOUNDS. To receive certification to conduct analyses for volatile organic compounds the laboratory shall do all of the following:

(a) Analyze reference samples which include volatile organic compounds provided by EPA; and

(b) Achieve quantitative results on the analyses performed under par. (a) that are within the acceptance limits listed in 40 CFR 141.24 (f) (17) (i) (C) and (D) and 40 CFR 141.24 (f) (17) (ii) (B); and

(d) Achieve acceptable results for at least 80% of the volatile organic chemicals; and

(e) Except for vinyl chloride, achieve a limit of detection of 0.0005 mg/L. For vinyl chloride, the laboratory shall achieve a limit of detection of 0.0003 mg/L.

(8) REQUIREMENTS FOR OTHER ORGANIC COMPOUNDS. To receive certification to conduct analyses for synthetic organic contaminants and total trihalomethanes, the laboratory shall:

(a) Analyze reference samples provided by EPA and meet the acceptance limits listed in 40 CFR 141.24 (h) (19) (i) (B).

(b) Achieve limits of detection as specified in 40 CFR 141.24 (h) (13) (ii) and (h) (18) or 10% of the MCL, whichever is greater.

Note: This publication is available for inspection at the offices of the Department of Natural Resources, the Secretary of State, and Revisor of Statutes. Copies are available from EPA, CERI, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268, 513–569–7562.

(9) NOTIFICATION. A laboratory certified under this chapter for category 18, safe drinking water, and analyzing a drinking water sample for analytes regulated under ch. NR 809, shall notify the facility immediately but no later than 48 hours if a test shows that a compliance sample exceeds the MCL for any regulated analyte.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; and recr. Register, November, 1992, No. 443, eff. 12–1–92; renum. (1) to (6) and (8) to be (2) to (7) and (1) and am. (1), (6), (7), r. (7), cr. (8), Register, April, 1994, No. 460, eff. 5–1–94; corrections made under s. 13.93 (2m) (b) 7., Stats., Register, April, 1994, No. 460; am. (7) (b), cr. (9), Register, February, 1996, No. 482, eff. 3–1–96.

NR 149.22 Requirements for effluent toxicity certification and registration. This section applies to those laboratories certified or registered under test category 20. The required quality control procedures along with the criteria and procedures for effluent toxicity testing are given in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 1st Edition," Wisconsin Department of Natural Resources, 1996.

Note: This publication is available for inspection at the offices of the Department of Natural Resources, the Secretary of State, and the Revisor of Statutes. Copies are available from the Department of Natural Resources, Bureau of Integrated Science Services, P.O. Box 7921, Madison, WI 53707.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; r. and recr. Register, November, 1992, No. 443, eff. 7–1–93; **am. Register, November, 1996, No. 491, eff. 12–1–96.**

NR 149.41 Laboratory evaluations. (1) The department shall conduct an on–site evaluation of each laboratory not more than once every 3 years unless there is reason to believe the laboratory is not in compliance with this chapter or if the laboratory requests an additional evaluation. The on–site evaluation shall be used to determine compliance with this chapter. The laboratory shall respond to the deficiencies cited in the evaluation report within 30 days. An unannounced follow–up evaluation may be performed after a notice of violation has been issued to verify that the deficiencies have been corrected.

(2) The department shall prepare an analysis of laboratory evaluation every year for review by the council. The council shall advise the department on the frequency and scope of evaluations necessary to determine compliance with this chapter.

(3) Before certification or registration may be granted, the laboratory shall meet the criteria and requirements specified in this chapter and be able to perform analyses in accordance with approved methods. Deficiencies identified during the initial laboratory evaluation shall be corrected before certification or registration can be issued.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; am. (1), Register, November, 1992, No. 443, eff. 1–1–93, cr. (3), eff. 7–1–93.

NR 149.42 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 analyte, group of analytes or test category. Contested case hearings for out of state laboratories regulated by this chapter shall be held in Madison, WI.

(a) *Causes for suspension of certification.* Causes for suspension include any of the following:

1. Failure to implement or comply with a quality control program as specified under s. NR 149.14.

2. Failure to follow approved methods.

3. Failure to maintain records as required in s. NR 149.06.

4. Failure to pay fees.

5. Conditions are present which render the laboratory temporarily incapable of performing analysis or analyses in the test category or categories.

6. A demonstrated incompetency which includes but is not limited to:

a. Failure of 3 consecutive reference samples for the same analyte or analyte group or failure to analyze the reference samples within the time limit specified in s. NR 149.13 (8) or (9). Suspension shall only be for the analyte, analyte group or test category in which inability to meet acceptance limits on reference samples or failure to analyze reference samples has been demonstrated.

b. Reporting data inaccurately.

7. Suspension of certification by another state if the grounds for which the suspension was issued are substantially equivalent to any of those listed in this subsection.

8. Conducting analysis for test categories for which certification has not been granted.

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

1. 'Fraudulent practices'. Fraudulent practices may include, but are not limited to any of the following:

a. Submitting reference sample results from another laboratory for compliance with s. NR 149.13.

b. Altering a certificate.

c. Falsification by the laboratory of analytical results, testing dates or any other information submitted to the department by the laboratory or another party.

2. Failure to pay fees.

3. Failure to submit requested records to the department.

4. Failure to allow the department or its representative to inspect the laboratory.

5. Failure to follow approved methods.

6. Failure to maintain records as required in s. NR 149.06.

7. A demonstrated incompetency which includes but is not limited to:

a. Chronic failure of reference samples, either by analyte group or as a whole.

b. Reporting data inaccurately.

c. Failure of 2 consecutive reference samples or failure to analyze the required reference samples for the safe drinking water test category. Revocation in the safe drinking water test category may be by analyte or analyte group.

8. Revocation of certification by another state if the grounds for which the revocation was issued are substantially equivalent to any of those listed in this subsection.

9. Failure to implement or comply with a quality control program as specified under s. NR 149.14

(c) *Causes for revocation of registration.* If the laboratory has falsified results or has materially and consistently failed to comply with the quality control procedures specified in s. NR 149.14, the laboratory's registration may be revoked by analyte or by test category or categories.

(d) *Procedure for suspension or revocation of certification or revocation of registration.* 1. An order suspending or revoking the 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.

2. 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 to the department within 30 days. 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). If a request 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.

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

(e) *Reapplication.* 1. A laboratory which has had its certification suspended may reapply for certification if the deficiencies that led to the suspension have been corrected in accordance with the timetable contained in the order and conditions for reapplication specified in the order have been met. The department shall consider the application complete if the laboratory:

a. Provides the department documentation which is acceptable to the department that demonstrates the conditions of the order have been met,

b. Pays required fees,

c. Has acceptable reference samples results when required under s. NR 149.13,

d. Submits a written request for reinstatement.

2. A laboratory which has had its certification or registration revoked may reapply for certification or registration if all of the following are completed:

a. The deficiencies that led to the revocation have been corrected,

b. Conditions contained in the order have been satisfied.

c. The time period for which the revocation is in effect has expired, and

d. The requirements of s. NR 149.07 are met.

(2) 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 605 or 630, may be referred by the department to the attorney general's office for enforcement.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; r. and recr. Register, November, 1992, No. 443, eff. 1–1–93; cr. (1) (a) 8., (b) 9., am. (1) (d) 2., r. and recr. (2), Register, April, 1994, No. 460, eff. 5–1–94; **corrections in (2) (a) made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.**

NR 149.43 Recognition of other certification or registration. (1) RECIPROCITY WITH LABORATORIES CERTIFIED OR REGISTERED BY OTHER GOVERNMENTS. The department may recognize the certification, registration, licensure or approval of a laboratory by another state or an agency of the federal government if the standards for certification, registration, licensure or approval are substantially equivalent to those established under this chapter. The department shall negotiate with and attempt to enter into acceptable agreements with federal agencies and agencies of other states for the purpose of reciprocal recognition of laboratory certification and registration under this chapter. The department may not recognize the certification, registration, licensure or approval of a laboratory by another state or an agency of the federal government unless that state or federal agency recognizes laboratories certified under this chapter. The department may accept the results of any tests conducted by a laboratory which it recognizes under an agreement. The department shall publish periodically a list of those agencies whose certifications, approvals or registrations it accepts. Any laboratory which is registered, certified or approved by any such agency may apply to the department to have the same recognized under this chapter.

(2) PRIVATE ORGANIZATION AGREEMENTS. The department may recognize the certification, accreditation or approval of a laboratory by a private nonprofit organization if the organization's standards for certification, accreditation or approval are substantially equivalent to those established under this chapter. The department may negotiate with and attempt to enter into acceptable agreements with private nonprofit organizations for the purpose of recognition under this subsection. The department shall publish periodically a list of those organizations whose certifications, accreditations or approvals it accepts. The department may accept

the results of any tests conducted by a laboratory that it recognizes under an agreement. Any laboratory that is certified, accredited or approved by an organization with which the department has an agreement may apply to the department to be recognized under this subsection.

(3) HEALTH AND SOCIAL SERVICES AGREEMENT. The department shall recognize the certification of a laboratory by the department of health and social services under s. 143.15, Stats., and shall accept the results of any test conducted by a laboratory certified to conduct that category of test under that section.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86; r. and recr. (1), (2), Register, April, 1994, No. 460, eff. 5–1–94.

NR 149.44 Discretionary acceptance. The department may accept on a case–by–case basis the results of a test in a specified test category even though the test was not conducted by a certified or registered laboratory. The department may charge a fee under s. 299.11 (5) (d), Stats., if it is necessary to verify the results of a test submitted under this section. The department may not accept data that do not meet the requirements established in this chapter. This section does not apply to monitoring required under ch. NR 809, where a certified laboratory is required.

History: Register, April, 1986, No. 364, eff. 5–1–86; am. (1), r. (2), Register, November, 1992, No. 443, eff. 12–1–92; correction made under s. 13.93 (2m) (b) 7, Stats., Register, April, 1994, No. 460; am. Register, February, 1996, No. 482, eff. 3–1–96; **correction made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.**

NR 149.45 Variances. (1) GENERAL. The department may, with the advice of the council, approve variances from non-statutory requirements of this chapter when it is determined that such variances are essential to department objectives or have no effect on the department's objectives. Before granting variances, the department shall take into account such factors as good cause, circumstances beyond the control of the laboratory, and financial hardship. A written summary of variances issued by the department shall be presented to the council annually.

(2) REQUEST FOR VARIANCE. A request for a variance shall be submitted in writing to the director, office of technical services, department of natural resources, as far in advance as the situation will permit. Each request for a variance shall contain the following:

(a) The name of the applicant or laboratory;

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

(c) An adequate description of the variance and the circumstances in which it will be used, including pertinent background information which is relevant to making a determination of justification; and

(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.

(3) APPROVAL OF VARIANCE. A letter of approval or denial of the variance shall be sent to the applicant. If the request is denied, the letter shall include reasons for the denial. A copy of each such written approval or denial shall be retained in the department's files.

History: Cr. Register, April, 1986, No. 364, eff. 5–1–86.