# Chapter NR 149

# LABORATORY CERTIFICATION AND REGISTRATION



## Subchapter I — General

**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. 144.95, Stats.

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

**NR 149.02 Applicability.** (1) Except as provided in subs. (2) and (3), the provisions of subchs. I, II and IV are applicable to laboratories applying for certifications or registrations and laboratories holding valid certification or registration, where department rules require laboratory tests to be done by a certified or registered laboratory.

(2) The provisions of subchs. I, III and IV are applicable to laboratories applying for certification and laboratories holding valid certifications for the analysis of samples for the safe drinking water program under ss. NR 109.12, 109.13, 109.14, 109.21, and 109.22.

(3) This chapter is not applicable 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.

Note: Administrative codes requiring analyses to be done by a certified or registered laboratory are: chs. NR 109, 110, 123, 131, 132, 140, 150, 157, 158, 180, 181, 182, 210, 211, 212, 219 and 347.

History: Cr. Register, April, 1986, No. 364, eff. 5-1-86; am. (1) and (2), Register, April, 1988, No. 388, eff. 5-1-88.

**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.

(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 St. Claire, Cincinnati, Ohio 45268, Revised 1983, including EPA-600 4-84-017, March, 1984.

(b) "Test Methods - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", EPA-600 4-82-057, Environmental Monitoring and Support Laboratory, 26 West St. Claire, Cincinnati, Ohio 45268, July, 1982.

(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, July, 1982, including update number 1, 1984.

(d) "Standard Methods for the Examination of Water and Wastewater", 16th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1985.

(e) "1984 Annual Book of ASTM Standards, Section 11.01 and 11.02, 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 St. Claire, 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.

(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 St. Claire, Cincinnati, Ohio 45268, March, 1979.

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

(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.

(29) "Spiked sample" means a duplicate sample to which a known amount of the analyte has been added to determine percent recovery.

(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.

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

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

**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 subch. III and certification shall be for each analyte within this test category.

**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. |
| 3. | Phosphorus | Total Phosphorus | Orthophosphate, Phosphorus |
| 4. | Physical | Total Suspended Solids | Total Solids, Dissolved Solids, Volatile Solids, Total Suspended Solids. |
| 5. | General I | Chloride | Alkalinity/Acidity, Chloride, Hardness, Sulfate |
| 6. | General II | Each analyte for which certification or registration is desired. | Chemical Oxygen Demand, Cyanide, Fluoride, Total Phenolic Compounds |
| 7. | General III | No reference sample | Bromide, Color, odor, Oil and Grease, Specific Conductance, Sulfide, Sulfite, Surfactants, Turbidity |
| 8. | General IV | No reference sample | Corrosivity, EP Toxicity, Ignitability, Reactivity, Total Organic Carbon, Total Organic Halide. |
| 9. | Metals I | Copper and Cadmium | Aluminum, Antimony, Barium, Beryllium, Bismuth, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Gold, Iridium, Lead, Lithium, Magnesium, Manganese, Molybdenum, Nickel, Osmium, Palladium, Platinum, Potassium, Rhodium, Ruthenium, Silicon, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Tungsten, Vanadium, Zinc, and Zirconium. |
| 10. | Metals II | Each analyte for which certification or registration is desired | Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Gold, Iridium, Lead, Lithium, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Osmium, Palladium, Platinum, Potassium, Rhodium, Ruthenium, Selenium, Silicon, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Tungsten, Vanadium, Zinc, and Zirconium. |

| No. | Test Category | Key Analyte | Analytes In Test Category (Includes forms of the given analytes) |
|---|---|---|---|
| 11. | Organics; Purgeable by Gas Chromatography or Gas Chromatography/Mass Spectrometer | Trichloroethene and Benzene | Purgeable Halocarbons, Purgeable Aromatics, Arolein, Acrylonitrile. |
| 12. | Organics; Base/Neutral Extractables by Gas Chromatography or Gas Chromatogrpahy/Mass Spectrometer | P,P'-DDT and Dieldrin | Phthalate Esters, Nitrosamines, Organochlorine Pesticides, Nitroaromatics, Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons, Base Neutral Extractable Pesticides (e.g., Atrazine, Cyanazine, Phorate, Linuran, and Butylate). |
| 13. | Organic; Acid Extractables by Gas Chromatography or Gas Chromatography/Mass Spectrometer | Pentachlorophenol | Phenolic Compounds. |
| 14. | Organics; Extractables by Liquid Chromatography | Naphthalene | Benzidines, Polynuclear Aromatic Hydrocarbons, Pesticides subject to Liquid Chromatography (e.g., carbofuran, oxamyl, and methomyl). |
| 15. | Organics; Acid Extractable Pesticides | 2,4-D | 2,4-D, 2,4,5-T, Picloram, Chloramben, and other acid extractable pesticides. |
| 16. | Pesticides not included in other test categories | 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). | Aldicarb, Ethylene Dibromide, and Glyphosate, and other pesticides. |
| 17. | Organic-Polychlorinated Biphenyls | PCB (Common Aroclor) | Polychlorinated Biphenyls. |
| 18. | 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. |
| 19. | Safe Drinking Water | Each analyte for which certification is desired. | Arsenic, Barium, Cadmium Chromium, Fluoride, Lead, Mercury, Nitrate as Nitrogen, Selenium, Silver, Sodium, Endrin, Lindane, Methoxychlor, Toxaphene, 2,4-D, 2,4,5, - TP, Total Trihalomethanes, and Corrosivity. |
| 20. | Any Single Analyte | That Analyte. | That Analyte. |

Note: The test category for metals I does not contain arsenic, mercury, or selenium. These metals along with all the metals in metals I can be found in metals II.

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

**NR 149.05 Fees.** (1) Fees for certification or registration and other listed items shall be as follows:

(a) Annual fee for all laboratories applying for certification or registration under this chapter - $150

(b) Annual fee for each test category except safe drinking water test category - $25

(c) Annual fee for safe drinking water test category - $300

(d) Late fee - $25

(e) On-site evaluation fee for certification or registration of out-of-state laboratories - Travel costs and travel time

(f) Discretionary acceptance - Actual cost of determining data quality

(g) Reference sample provided by department or its agent - Actual cost

Note: There will be no reciprocity fee for the evaluation of other certification programs.

(2) REFUNDS. Fees are not refundable.

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

(4) FEE REVISION. Any fee change shall be based on a demonstrated need for revision to support the level of effort in the program and shall be reviewed by the council before being proposed as a rule amendment.

(5) PRORATED FEES. For laboratories applying for initial certification or registration, fees shall be prorated at ½ of the annual fee if the laboratory is applying after January 1.

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

**NR 149.06 Records.** (1) The following records shall be retained by the certified or registered laboratory 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) A record of samples processed so that any sample may be traced back to the analyst, date and method used.

(b) Quality control date for spikes, duplicates, reagent blanks, blind standards, reference samples, calibration standards and known standards.

(c) Quality control limits for each parameter.

(d) Information on maintenance of laboratory instruments.

(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 depart-

ment 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, 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) Each subsidiary or branch laboratory shall independently comply with this section.

Note: Chapter NR 109, safe drinking water program contained in subch. III, 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.

**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 fee prescribed in s. NR 149.05.

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

(b) Specify the test catagories 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. 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 or 149.21.

(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.

(f) Submit to the department acceptable results on reference samples for test categories requiring reference samples.

(2) EVALUATION. For a laboratory to become certified for the safe drnking water test category, successful completion of an on-site laboratory evaluation is required.

(3) 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 described in subs. (1) and (2).

(4) RENEWAL OF CERTIFICATION OR REGISTRATION. (a) Certifications and registrations shall be renewed prior to July 1 of each year. Prior to July 1 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 reference sample analysis and results required under s. NR 149.13 (4) shall be completed prior to renewal.

(c) A late fee shall be charged for those laboratories submitting fees after August 1.

(5) SUBSIDIARY OR BRANCH LABORATORIES. One application shall be completed by a laboratory having subsidiary or branch laboratories. Subsidiary or branch laboratories may apply for independent certification or registration. The application shall specify the location and supervisor of each subsidiary or branch laboratory which will be analyzing samples and submitting results to the department, where test results are required to be from a certified or registered laboratory.

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

## Subchapter II - Requirements for Certification or Registration

**NR 149.11 Methodology for laboratory analysis, sample collection, sample preservation, and holding time.** (1) The analytical methodology used for a specific test shall be the analytical methodology required by applicable state and federal laws. If the methodology for the test is not specified by state or federal laws, then a method from an authoritative source shall be used. The method from the authoritative source shall be appropriate for the test and the sample matrix.

Note: Analytical methodologies required by state law are in chs. NR 219 and 181. Those required by federal law are in 40 CFR 136 and 261.

(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 law are in chs. NR 218, 140 and 181.

(3) Sample preservation procedures and holding times required by state and federal laws shall be followed. If the sample preservation proce-

dures and holding times are not required by state or federal laws, the sample preservation procedures and holding times established in the analytical methodology shall be followed. If the methodology does not establish sample preservation procedures or holding times, procedures in the authoritative sources shall be followed. If the sample is not preserved or if the holding time of the sample exceeds the holding time required under this section, the laboratory shall report this fact with the results.

Note: Sample preservation procedures and holding times required by federal law are in 40 CFR 136.

(4) The test results from a certified or registered laboratory shall be reported as follows:

(a) Concentrations less than the limit of detection shall be reported as not detected, and the limit of detection shall be stated.

(b) Concentrations greater than the limit of detection shall be reported.

(c) If requested by the department, the limit of quantitation for an analyte shall be determined. If the limit of quantitation of an analyte has been requested for a particular sample matrix, the limit of quantitation shall be reported with all concentrations that are above the limit of detection and below or equal to the limit of quantitation.

(5) The limit of quantitation and limit of detection may be determined in accordance with s. NR 149.03 (5) (j).

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

**NR 149.12 Alternate methodology.** (1) Alternate methodologies may be used if the department or EPA has granted an approval for their use. If EPA has approved the methodology, a copy of such approval shall be provided to the department. Department approval does not constitute approval by EPA. The department may waive some or all of the requirements of sub. (2) where the department finds the information is available elsewhere.

Note: State or federal laws may require EPA approval of the alternate methodology. Alternate methodology approval by EPA is required by state law in ch. NR 219 and by federal law in 40 CFR 136, 141, and 260.

(2) In order to request department approval for an alternate methodology, the following information shall be provided to the department:

(a) A detailed description of the alternate methodology.

(b) Comparability data shall be generated and reported for samples from each type of discharge or water supply for a maximum of 5 sources. For each source, a minimum of 3 samples shall be collected which are representative of the expected concentration range of the analyte of interest or changes in the sample matrix. Each sample shall be analyzed 8 times, 4 replicates each by the proposed alternate methodology and an approved methodology. Cite the approved methodology used. If the expected concentration would be below the detection limit, then the sample shall be spiked to raise the concentration to a detectable level.

(c) Results of 7 spiked samples, for those tests where spiking is appropriate, from the alternate methodology. The spiking compound shall be the same as used in the approved methodology.

Note: Spiked sample analysis is not appropriate for alkalinity, color, conductivity, oil and grease, biochemical oxygen demand, carbonaceous biochemical oxygen demand, pH, reactivity, solids, turbidity, corrosivity, ignitability, sulfide, sulfite, and certain other tests.

(d) Justification for the use of the alternate methodology and the sample matrices for which the alternate methodology will be used.

(e) Results from the analysis of 2 reference samples analyzed by the laboratory applying for approval of the alternate methodology, utilizing the proposed alternate methodology.

(3) Laboratories submitting test results utilizing an alternate methodology may request confidentiality for the description of the method. Confidentiality determinations shall be made consistent with s. 144.95 (7) (b) 3.b., Stats.

Note: Procedures set forth in ch. NR 2 shall be followed for application and determination of confidentiality.

(4) Upon review of the information submitted by the laboratory under sub. (2), a determination of the approvability of the methodology shall be made. The following criteria shall provide the basis for the determination:

(a) Whether the precision and accuracy obtained through use of the alternate methodology equals or exceeds that obtained through use of the accepted methodology; and

(b) Whether the methodology meets the requirements of state and federal law on alternate methodology, where applicable.

(5) Once the determination of approvability has been made by the department, a recommendation with the information in sub. (2) shall be forwarded to EPA for a ruling on approval, if required under state or federal law. If approval by EPA is not required, the department shall approve or disapprove the methodology within 60 days of receipt of the information required under sub. (2). If approval is denied, the reasons for denial shall be provided to the applicant.

(6) Authoritative sources not listed under s. NR 149.03 (5), but approved by the department, with the recommendation of the council, may be used by certified or registered laboratories.

(7) The department may permit the use of a revised methodology consistent with new or revised editions or standards established by technical societies and organizations on a case-by-case basis without requiring compliance with the procedure in this section.

(8) The department shall accept data obtained through use of the alternate methodology after the method has been approved under this section.

(9) The department shall maintain a list of approved nonconfidential alternate methodologies.

(10) The department may waive any portion of any procedure prescribed in the accepted methodology on a case-by-case basis if the labora-

tory seeking this waiver established sufficient reasons for the waiver and that the waiver does not adversely affect the purpose for which the test is conducted.

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

**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. Reference sample results shall be reported to the department. 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) Each subsidiary or branch laboratory shall analyze reference samples for each test category for which the subsidiary or branch laboratory conducts tests at that location.

(3) The department may provide each certified laboratory with a maximum of 3 reference samples per year for each of the test categories for which the laboratory seeks certification, or the laboratory may procure its own reference samples. For a registered laboratory one reference sample per year may be provided for each test category for which the laboratory seeks registration or the laboratory may procure its own reference samples. Reference samples shall be obtained from the department or a source approved by the department. Reference samples shall be provided if requested. The cost of providing this service shall be billed to the laboratory requesting the reference samples. The following is a list of approved reference samples sources:

(a) United States environmental protection agency, discharge monitoring quality assurance study reference samples.

(b) United States environmental protection agency, performance evaluation reference samples.

(c) State laboratory of hygiene.

(d) Other reference sample providers are acceptable on a case-by-case basis. Criteria for approving other providers are:

1. The means of calculating the acceptance limits shall be at least as stringent as the calculation of acceptance limits for the department's reference samples.

2. 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.

3. 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, samples provided by the department or a source approved under sub. (3) (d) shall be analyzed and reported to the department between February 1 and March 31 of each year. 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). Additional reference sample results may be submitted after March 31.

(5) For reference samples not issued by the department, a given laboratory's results are acceptable if they are within the reference sample provider's acceptance limits.

(6) For reference samples provided by the department or an agent of the department, a given laboratory's results are acceptable if they are within 2.78 standard deviations of the mean after the exclusion of outlying results. The calculation of the mean and standard deviation will be based on the results of all laboratories submitting reference sample results. Outliers shall be determined by the American society for testing and materials standard practice E 178-80 or D 3856-80.

Note: Copies of these standard practices are available for inspection at the offices of the department of natural resources, the secretary of state, and the revisor of statutes. Copies may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

(7) If a laboratory does not meet the acceptance limits of the reference sample provider or the department, 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 to determine if the laboratory is following its quality control program.

(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 3 reference samples.

(11) For test categories 16 and 18, no reference sample is required. The laboratory shall demonstrate, upon application for certification or registration, acceptable precision and percent recovery based on duplicate analysis and spiked sample analysis. The following information shall be submitted:

(a) A detailed description of the methodology.

(b) Results of 15 samples analyzed in duplicate using the above submitted methodology. Samples chosen for duplicate analysis shall be representative of those types typically analyzed by the laboratory. The samples shall include the range of expected concentrations. If the expected concentration would be below the detection limit, the samples shall be spiked to raise the concentration to a detectable level.

(c) Results of 7 spiked samples and the calculated spike recovery.

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

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

(2) Each subsidiary or branch laboratory shall independently comply with this section.

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

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

(b) A known standard analyzed or a calibration done on each analysis day. The instrument response for the known standard shall be within the pre-established limits under par. (c).

(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, 9, 10 and for total organic carbon, total organic halide, chloride, hardness, and sulfate, the pre-established limit shall be $\pm 10\%$.

2. For test categories 11, 12, 13, 14, 15, 16, 17, and 18, the pre-established limits shall be $\pm 15\%$.

3. There is no requirement to analyze a known standard for alkalinity acidity, corrosivity, EP toxicity, ignitability reactivity, gravimetric tests, titratimetric tests, and test categories 4 and 7.

4. For test category 1, a known standard shall be analyzed after the analysis of 20 samples or once a week. The limits on this quality control check shall be as established in an authoritative source.

5. For test category 20 the pre-established limit shall be appropriate for the test.

(d) At least one reagent blank shall be analyzed on each analysis day, for those tests for which reagent blanks are appropriate. For certain tests, a nonreacted sample may be used as a blank.

Note: Reagent blanks are not appropriate for certain tests such as alkalinity acidity, conductivity, hardness, ignitability, and pH. Nonreacted sample blanks are appropriate for certain colormetric and turbidimetric tests such as the sulfate turbidimetric test, silica molybdosilicate test, and the phosphorus ascorbic acid test.

(e) A duplicate sample shall be run after the analysis of 10 samples. For those methodologies which require that the sample bottle be extracted, duplicate samples shall be taken in the field to insure representative samples.

(f) Spiked samples shall be analyzed except when the method of standard addition is used. The spiking of the sample shall be done before any extraction or digestion. The frequency of spiked analysis shall be:

1. As required in the authoritative sources for test categories 11 to 18, 20, total organic halide and total organic carbon. If no frequency is given in the authoritative source, then the frequency shall be after the analysis of 10 samples.

2. After the analysis of 20 samples, at a minimum, for test categories 2, 3, 6, 9, 10, and for chloride, hardness, sulfate, and bromide.

3. No spiked analysis is required for test categories 1 and 4 and for alkalinity acidity, color, EP toxicity, pH, oil and grease, specific conductance, sulfide, sulfite, turbidity, corrosivity, ignitability, reactivity, and gravimetric tests, or tests where appropriate standards are not available for spiking.

Note: Spiked samples for organics should be spiked with representative organic analytes for each analyzed extraction. The representative organic analytes should be chosen on the basis of the organic analytes which are identified within a permit, and organic analytes which are typically found in that type of sample.

(g) Quality control limits for duplicate sample and spiked sample analysis shall be calculated using a method from an authoritative source. For laboratories with less than 30 quality control results within 12 months, the laboratory may set quality control limits based on information given in the authoritative sources, or laboratory experience, or the experience of other laboratories.

(h) If the results of known standards or duplicates exceed 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 back to the last acceptable quality control check. 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) A blind standard shall be analyzed, if available for that analyte, every 4 months for each analyte in test categories 1 to 10, 16, and 20, if the analyte was analyzed during the previous 4-month period. A blind standard shall be analyzed every 4 months for one analyte in each test category in test categories 11 to 15, 17, and 18, if an analyte within those test categories was analyzed during the previous 4-month period. 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.

(k) Where duplicate, 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.

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

(5) If it has been determined that an organic analyte is present in a sample, the laboratory shall inform the data user if the results have been confirmed by a second analysis with a different methodology.

History: Cr. Register, April, 1986, No. 364, eff. 5-1-86; am. (3) (c) 4., Register, April, 1988, No. 388, eff. 5-1-88.

## Subchapter III - Requirements and Administrative Procedures for the Safe Drinking Water Test Category

Note: This subchapter applies only to laboratories applying for certification in the safe drinking water test category for the purpose of analyzing samples for the safe drinking water program under ch. NR 109.

**NR 149.21 Methodology.** The analytical methodology used for the safe drinking water test category shall comply with state and federal law. The laboratory instrumentation shall meet the specifications of the required methodology.

Note: A listing of approved methodologies is maintained by the department as specified by ch. NR 109 and this chapter. In addition federal law requirements for methodology can be found in 40 CFR 141 and 143.

(1) SAMPLE COLLECTION, HANDLING, AND PRESERVATION. The following procedures shall be used for sample collection, handling and preservation:

(a) The sample shall be representative of the potable water system. The water tap shall be sampled after maintaining a steady flow for 2 or 3 minutes to clear the service line. The tap shall be free of aerator, strainer, hose attachment, or water purification devices.

(b) The sample report form shall be completed immediately after collection with location, date and time of collection, collector's name, and any special remarks concerning the sample.

(c) The required type of sample container and the required preservation for each inorganic chemical analytes are listed in Table 2.

(d) All samples shall be analyzed within the maximum holding times listed in Table 2. Where these maximum holding times cannot be met, the sample shall be discarded and resampling shall be required.

(e) The required type of sample container and the required preservative for the organic chemical analytes are listed in Table 3.

(f) When sampling chlorinated waters for total trihalomethane analysis, sodium thiosulfate or sodium sulfite shall be added to the empty sample bottles prior to shipping to the sampling site.

(g) The total trihalomethane bottles shall be filled in such a manner that no air bubbles pass through the sample as the bottle is filled. The bottle shall be sealed so that no air bubbles are entrapped in it. The hermetic seal on the sample bottle shall be maintained until analysis.

(h) All organic samples shall be analyzed within the maximum holding times listed in Table 3. Where these maximum holding times cannot be met, the sample shall be discarded and resampling shall be required.

(i) Sodium samples shall be collected in plastic or glass containers. Samples shall be preserved by the addition of concentrated nitric acid to achieve pH of less than 2; analyses shall be performed within 6 months of collection.

(j) Alkalinity samples shall be collected in plastic or glass, and shall be preserved by cooling to 4°C. Analysis shall be performed within 14 days of collection.

(k) Calcium samples shall be collected in plastic or glass containers, cooled to 4°C and shall be preserved by the addition of concentrated nitric acid to achieve a pH of less than 2. Analyses shall be performed within 6 months of collection.

(l) Chloride samples shall be collected in plastic or glass containers. Samples need not be preserved. Analyses shall be performed within 7 days of collection.

(m) Sulfate samples shall be collected in plastic or glass containers, and shall be preserved by cooling to 4°C. Analyses shall be performed within 7 days of collection.

(n) Total dissolved solids samples shall be collected in plastic or glass containers, and shall be preserved by cooling to 4°C. Analyses shall be performed within 7 days of collection.

Note: Sample collection methods required by federal law are in 40 CFR 141.30 Appendix C.

(2) ALTERNATE METHODOLOGY. In order to obtain approval to use an alternate methodology, the procedures in s. NR 149.12 shall be followed.

TABLE 2

Sample Collecting, Handling, and Preservation for Inorganic Analytes[1]

| Analyte | Preservation[2] | Containers[3] | Maximum Holding Time[4] |
|---|---|---|---|
| Arsenic | Conc $HNO_3$ to pH < 2 | P or G | 6 months |
| Barium | Conc $HNO_3$ to pH < 2 | P or G | 6 months |
| Cadmium | Conc $HNO_3$ to pH < 2 | P or G | 6 months |
| Chromium | Conc $HNO_3$ to pH < 2 | P or G | 6 months |
| Fluoride | None | P | 1 month |
| Lead | Conc $HNO_3$ to pH < 2 | P or G | 6 months |
| Mercury | Conc $HNO_3$ to pH < 2 | G | 38 days |
| | | P | 14 days |
| Nitrate | | | |
| Chlorinated supplies | Cool 4°C | P or G | 28 days |
| Nonchlorinated supplies | Conc $H_2SO_4$ to pH < 2 | P or G | 14 days |
| Selenium | Conc $HNO_3$ to pH < 2 | P or G | 6 months |
| Silver | Conc $HNO_3$ to pH < 2 | P or G | 6 months |

1 If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and so notify the authority requesting the analyses.

2 If $HNO_3$ cannot be used because of shipping restrictions, the sample may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with concentrated $HNO_3$ to pH < 2. At the time of analysis, the sample container should be thoroughly rinsed with 1:1 $HNO_3$; washings should be added to the sample.

3 P = Polyethylene; G = Glass.

4 In all cases, samples should be analyzed as soon after collection as possible.

TABLE 3

**Sample Collection, Handling, and Preservation for Organic Analytes[1]**

| Analyte | Preservation | Containers | Maximum Holding Time[2] |
|---|---|---|---|
| Chlorinated hydrocarbons | Refrigerate at 4°C as soon as possible after collection | Glass with foil or Teflon-lined cap | 14 days[3] |
| Chlorophenoxys | Refrigerate at 4°C as soon as possible after collection | Glass with foil or Teflon-lined cap | 7 days[3] |
| Total Trihalomethane | Sodium thiosulfate or sodium sulfite | Glass with Teflon-lined septum[4] | 14 days |

1 If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and so notify the authority requesting the analyses.

2 In all cases, samples should be analyzed as soon after collection as possible.

3 Well-stoppered and refrigerated extracts can be held up to 30 days.

4 All samples shall be collected in duplicate.

TABLE 4

Approved Methods

| Measurement | Methodology | EPA[1] | ASTM[2] | SM[3] | Other |
|---|---|---|---|---|---|
| Free Chlorine residual | Colorimetric or titrimetric DPD | - | - | 409E or F | - |
| | Colorimetric syringaldzine | - | - | - | 408G[4] |
| | Amperometric Titrimetric | - | - | - | 409C[4] |
| pH | Potentiometric | 150.1 | D1293-78A or B | 424 | - |

1 "Methods of Chemical Analysis of Water and Wastes", EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268 (EPA-600/4-79-020), March, 1979. Available from ORD Publications, ERI, EPA, 26 West St. Claire, Cincinnati, Ohio 45268.

2 1980 Annual Book of ASTM Standards, Part 31 Water, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103. Available from American Society for Testing and Materials.

3 "Standard Methods for the Examination of Water and Wastewater", 14th Edition, American Public Health Association, American Water Works Asssociation, Water Pollution Control Federation, 1975.

4 "Standard Methods for the Examination of Water and Wastewater", 15th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1980.

**Note:** Copies of these publications are available for inspection at the offices of the Department of Natural Resources, the Secretary of State, and Revisor of Statutes. Copies of the methodology referenced in footnote 3 and 4 are available from the Department of Natural Resources, Office of Technical Services, P.O. Box 7921, Madison, WI 53707.

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

**NR149.22 Requirements for fluoride, chlorine residual, pH, and turbidity.** (1) FLUORIDE. Fluoride analyses required under s. NR 109.70 need not be performed by a certified laboratory, but may be performed by any person if such person adhere to the following requirements in his or her analyses:

(a) The specifications of s. NR 109.70 (1) (b) and (c) shall be followed.

(b) The requirements of Table 2 concerning preservation of the sample and type of sample container shall be followed.

(c) The methodology specified by ch. NR 109 shall be used.

(2) FREE CHLORINE RESIDUAL. Free chlorine residual and total chlorine residual analyses required under s. NR 109.70 need not to be done by a certified laboratory, but may be performed by any person if such person adheres to the following requirements in his or her analyses:

(a) Samples may not be preserved for later analysis. All analyses shall be made as soon as practicable, but not later than one hour after sample collection.

(b) Polyethylene or glass containers shall be used for sample collection.

(c) The methodology specified in Table 4 shall be used.

(d) Blind standards shall be analyzed semi-annually and those records shall be maintained for 3 years.

(3) ANALYSIS FOR PH. Analyses for pH required under s. NR 109.14 need not be done by a certified laboratory, but may be performed by any person if such person adhere to the following requirements in his or her analyses:

(a) Samples may not be preserved for later analysis. All analyses shall be made as soon as practicable, but no later than one hour after sample collection.

(b) Polyethylene or glass containers shall be used for sample collection.

(c) The methodology specified in Table 4 shall be used.

(d) Analysis shall be performed with a pH meter.

(e) Blind standards shall be analyzed semi-annually and those records shall be maintained for 3 years.

(f) pH meters shall be calibrated prior to use with fresh standard buffers at pH 7.0 and at the pH appropriate for the samples being tested.

(4) TURBIDITY. Turbidity analyses as required under s. NR 109.41 need not be done by a certified laboratory, but may be performed by any person if such person adheres to the following requirements in his or her analyses:

(a) Samples may not be preserved for later analysis. All analyses shall be made as soon as practicable, but no later than one hour after sample collection.

(b) Polyethylene or glass containers shall be used for sample collection.

(c) The methodology shall be that specified in s. NR 109.41 (2).

(d) Blind standards shall be analyzed semi-annually and those records shall be maintained for 3 years.

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

**NR 149.23 Reference samples.** (1) A reference sample shall be analyzed for each test parameter within the safe drinking water test category for which certification is requested.

(2) Reference samples for this test category shall be obtained from the department, EPA, or an agent of the department. Acceptance limits for the test results on the reference samples shall be those established by the EPA.

(3) If more than one concentration of a particular parameter is provided, the laboratory shall satisfactorily analyze all concentrations, except where otherwise stated. After failure to meet the acceptance limits on one reference sample, a laboratory may request quality control samples and technical assistance from the department to help identify and resolve the problem.

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

**NR 149.24 Quality assurance.** (1) GENERAL. (a) The laboratory shall prepare and follow a written quality assurance plan. Various documents may be incorporated into the quality assurance plan by reference. All quality assurance data shall be available for inspection. This plan shall include:

1. Sampling procedures.

Note: Acceptable sampling procedures are available from the Department of Natural Resources, Bureau of Water Supply, P.O. Box 7921, Madison, WI 53707.

2. Sample handling procedures. Specify procedures used to maintain integrity of all samples, i.e., tracking samples from receipt by laboratory through analysis to disposal. Samples likely to be the basis for an enforcement action may require special safeguards.

3. Instrument or equipment calibration procedures and frequency of their use.

4. Analytical methodology. The step-by-step procedures used to analyze test samples.

5. Data reduction, validation and reporting. Conversion of raw data to final results. This includes what procedures are used to insure accuracy of data during transcription and calculations. It shall include the procedures and format for reporting data to utilities, the department, and EPA.

6. Description of the types of internal quality control checks and frequency of their use.

7. Preventive maintenance procedures and schedules. This shall include what routine maintenance is done and at what frequency.

8. Specific routine procedures used to determine data precision and accuracy for each analyte measured. Precision is based on the results of duplicate analyses. Accuracy is determined by spike sample analyses.

9. Corrective action contingencies. This shall include laboratory response to unacceptable results from analysis of reference samples and internal quality control checks.

(b) A manual of analytical methods shall be available to the analysts.

(c) Standardized class "S" weights shall be available at the laboratory to make periodic checks on balances.

(d) Chemicals shall be dated upon receipt of shipment and replaced as needed and before the shelf life has been exceeded.

(2) INORGANIC ANALYTES. (a) A calibration curve composed of a minimum of a reagent blank and 3 calibration standards covering the concentration range of the samples shall be prepared. At least one of the calibration standards shall be at or below the maximum contaminant level.

(b) For each day on which analyses are performed, the standard curve shall be verified by use of a reagent blank and one known standard within the range of the calibration curve. This verification shall be done for every 20 samples. Each known standard shall be within $\pm$ 10% of original curve.

(3) ORGANIC ANALYTES. (a) For each day on which pesticide or phenoxyacid analyses are initiated, or trihalomethane reagent water is prepared, a reagent blank shall be analyzed with the same procedures used to analyze samples.

(b) A minimum of 3 calibaration standards shall be analyzed each day to calibrate the analytical system. If the laboratory can demonstrate that the instrument response is linear and passes through zero, this practice may be reduced to one calibration standard per day, providing the response of the standard is within $\pm$ 15% of previous calibrations.

(c) Each quarter, the laboratory shall analyze blind standards obtained from the EPA for each parameter. If the acceptance limits established by EPA are not met, corrective action shall be taken and documented.

(d) The laboratory shall analyze the trip blank if trihalomethanes are detectable in the samples. If reportable levels of trihalomethanes are demonstrated to have contaminated the trip blank, the results shall be qualified and resampling is necessary.

(e) The laboratory shall analyze 10% of all samples for total trihalomethane in duplicate. A continuing record of results and subsequent actions taken shall be maintained.

(f) The laboratory shall analyze a total trihalomethane laboratory known standard each day it analyzes samples for total trihalomethane. If errors exceed 20% of the true value, all trihalomethane results since the previous successful tests are unacceptable. Samples shall be reanalyzed if it is determined that there has been no calculation or procedural error.

(g) Each time the total trihalomethane analytical system undergoes a major modification or prolonged period of inactivity, the precision of the system shall be demonstrated by the analysis of duplicate laboratory known standards.

(h) Laboratories that analyze for total trihalomethane by liquid-liquid extraction shall demonstrate that raw source waters do not contain interferences under the chromatographic conditions selected.

(i) If a mass spectrometer detector is used for total trihalomethane analysis, the mass spectrometer performance tests described in Table 5 in s. NR 149.26 (3) (c) 3. using p-Bromofluorobenzene shall be conducted once during each 8 to 12 hour work shift. Records of satisfactory performance and corrective action shall be maintained.

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

**NR 149.25 Action response to laboratory results.** When laboratory results indicate that a maximum contaminant level of any parameter has been exceeded by a public water supply, the public water supply shall be notified as soon as possible, but in any event within 48 hours, of the sample result. Maximum contaminant levels are contained in ch. NR 109.

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

**NR 149.26 Laboratory evaluation.** (1) An on-site evaluation is required once every 3 years and upon application for those laboratories applying for certification in the safe drinking water test category. Before certification may be granted, the laboratory shall meet the criteria and requirements specified in this subchapter for the safe drinking water test category.

(2) The on-site evaluation shall be conducted at a mutually agreeable time.

(3) Only those instruments that are needed to analyze for the parameters for which the laboratory is being certified are required, but those instruments shall meet the following minimum specifications:

(a) *General.* 1. Analytical balance. The analytical balance shall have a sensitivity of at least 0.1 mg. The balance shall be seated on a steady base to prevent interference due to vibration and shall be protected from interference due to air currents.

2. Magnetic stirrer. The magnetic stirrer shall have variable speed, with the stirring bar coated with inert material.

3. pH meter. The pH meter shall have accuracy of $\pm$ 0.05 units and scale readability of $\pm$ 0.1 units.

Note: Laboratories purchasing a new pH meter are strongly advised to purchase one capable of functioning with specific ion electrodes.

4. Conductivity meter. The conductivity meter shall be suitable for checking distilled water quality. The meter shall be readable in ohms or mhos, have a range from 2 ohms to meg-ohms or equivalent micromhos $\pm$ 1%.

5. Hot plate. Hot plates shall be large or small hot plates with selectable temperature controls for safe heating of laboratory reagents.

6. Refrigerator. The refrigerator shall be a standard kitchen type domestic, commercial, or laboratory grade refrigerator for storage of aqueous reagents and samples.

7. Drying oven. The drying oven shall be a gravity or mechanical convection drying oven with selectable temperature control from room temperature to 180°C (±2°) or higher.

8. Thermometer. The thermometer shall be a good grade mercury-filled centigrade thermometer with 1°C or finer subdivisions calibrated to 180°C or higher.

(b) *Inorganic analytes.* 1. Spectrophotometer. The spectrophotometer shall have a usable wavelength range of 400 to 70-0 nm, a maximum spectral bandwidth of no more than 20 nm, and a wavelength accuracy of ± 2.5 nm. The spectrophotometer shall be capable of using several sizes and shapes of absorption cells providing a sample path length from approximately 1 to 5 cm.

2. Filter photometer (abridged spectrophotometer). The filter photometer shall be capable of measuring radiant energy in range of 400 to 700 nm. Relatively broad bands (10 to 75 nm) of this radiant energy are isolated by use of filters or other isolation device at or near the maximum absorption of the colorimetric methods. The photometer shall be capable of using several sizes and shapes of absorption cells providing a sample path length varying from approximately 1 to 5 cm.

3. Specific ion meter. The specific ion meter shall be readable and accurate to ± 1mV.

4. Electrodes. The electrodes shall be pH electrodes, specific ion electrodes and reference electrodes as specified by the individual method.

5. Stirred water bath. The stirred water bath shall operate up to 100°C.

6. Automated analysis systems. Exact equipment used is specified by the individual method.

7. Arsine generator and absorption system. The arsine generator and absorption system shall be a Gutzeit generator or equivalent used in conjunction with an absorber tube or assembly.

8. Atomic absorption spectophotometer. The atomic absorption spectrophotometer shall be a single-channel and single or double beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wave length range of at least 190 to 800 nm.

a. An appropriate readout system that has a response time capable of measuring the atomic absorption signal generated is required. This includes the capability to detect positive interference on the signal from intense nonspecific absorption. In furnace analysis, a strip chart recorder shall be used for verification of adequate background correction if a CRT video readout or hard copy plotter is not available. The recorder shall have a chart width of 10 inches or 25 cm, full scale response time of 0.5 second or less, 10- or 100-mV input to match the instrument and variable chart speeds of 5 to 50 cm/min. or equivalent.

b. Commercial grade acetylene is acceptable. Air may be supplied from a compressed airline, a laboratory compressor, or from a cylinder of compressed air. Reagent grade nitrous oxide is also required for certain analyses. Standard, commercially available argon or nitrogen are required for furnace work, and hydrogen is required for the flame hydride systems.

The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

c. The burner recommended by the particular instrument manufacturer and consistent with the approved method shall be used. For certain elements, the nitrous oxide burner is required.

d. Single element lamps are preferred but multi-element lamps may be used. Electrodeless discharge lamps may also be used.

e. Any furnace device capable of reaching the specified temperatures is satisfactory.

f. Microliter pipets with disposable tips are acceptable. Sizes may range from 5 to 100 microliters as required. Pipet tips which are white in color and do not contain CdS may be used.

g. A background correction system or provision for subsequent analysis using a nonabsorbing line is required for furnace analysis.

h. Separatory funnels of 250 mL, or larger, are required for extraction with organic solvents.

i. Any gaseous hydride system is acceptable to use in conjunction with an atomic absorption spectrophotometer equipped for direct aspiration analysis.

9. Mercury cold vapor analyzer. A commercially available vapor mercury analyzer may be substituted for the equipment listed below.

a. A standard 10 cm quartz cell with end windows or a 11.5 cm plexiglass cell with an inner diameter of 2.5 cm.

b. A peristaltic pump with an air flow of one liter per minute.

c. A flowmeter capable of measuring an air flow of one liter per minute.

d. An atomic absorption spectrophotometer equipped with a mercury hollow cathode lamp.

e. A straight glass frit having a coarse porosity.

f. A 6-inch drying tube containing 20 grams of magnesium perchlorate or a heating device is required to prevent consolidation of moisture.

(c) *Organic analytes.* 1. Gas chromatograph. The gas chromatograph shall be a commercial or custom-designed gas chromatograph (GC) with a column oven capable of isothermal temperature control $\pm 0.2°C$ to at least 220°C. Additional required accessories and specifications are listed below by methodology.

a. For chlorinated hydrocarbons, the gas chromatograph shall be equipped with a glass lined injection port suitable for chlorinated hydrocarbon pesticides with a minimum of decomposition, and equipped with either an electron capture, microcoulometric titration, or electrolytic conductivity detector.

b. For chlorophenoxys, the gas chromatograph shall be equipped with a glass lined injection port and either an electron capture, microcoulometric titration, or electrolytic conductivity detector.

c. For total trihalomethanes by the purge and trap methodology, the gas chromatograph shall be temperature programmable from 45° to 220°C at 8°C/min and equipped with either microcoulometric titration or electrolytic conductivity detector.

d. For total trihalomethanes by the liquid/liquid extraction methodology, the gas chromatograph shall be equipped with a linearized, frequency modulated electron capture detector.

e. For total trihalomethanes by the gas chromatography/mass spectrometry method the gas chromatograph, which shall be temperature programmable, shall be interfaced to the mass spectrometer with an all-glass enrichment device and an all-glass transfer line. Mass spectral data shall be obtained with electron-impact ionization at a nominal electron energy of 70EV. The mass spectrometer shall produce a spectrum that meets all criteria in Table 5 when 50 ng or less of p-bromofluorobenzene (BFB) is introduced into the gas chromatograph. An interfaced data system is necessary to acquire, store, reduce and output mass spectral data. The data system shall be equipped with software to acquire and manipulate data for only a few ions that were selected as characteristic of trihalomethanes and the internal standard or surrogate compound.

2. Recorder for gas chromatograph. A strip chart recorder having a chart width of 10 in or 25 cm, a full scale response time of one second or less, 1-mV (-0.05 to 1.05) signal to match the instrument and a chart speed of 0.25 to 0.5 in/min or equivalent is required.

3. Purge and trap system. A purge and trap system which is of a commercial or custom-designed system containing 3 separate elements is required. When used with a compatible gas chromatograph, the assembly shall be able to detect 0.5 ug/L of each of the individual trihalomethanes and measure them with a reproducibility not to exceed 8% relative standard deviation at 20 ug/L.

**TABLE 5**

p-Bromofluorobenzene key ions
and Ion Abundance Criteria

| Mass | Ion Abundance Criteria |
|---|---|
| 50 | 15 to 40% of mass 95 |
| 75 | 30 to 60% of mass 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 to 9% of mass 95 |
| 173 | less than 2% of mass 174 |
| 174 | Greater than 50% of mass 95 |
| 175 | 5 to 9% of mass 174 |
| 176 | greater than 95% but less than 101% of mass 174 |
| 177 | 5 to 9% of mass 176 |

a. The purging device shall be designed for a 5 mL sample volume.

b. The trapping device shall be capable of retaining purged trihalomethanes at room temperatures.

c. The desorber assembly shall be capable of heating the trapping device to 180°C in one minute with less than 40°C overshoot.

4. Kuderna-Danish glassware each consisting of 3 ball Snyder column, evaporative flask, and calibrated ampul is required.

5. A water bath with electric or steam heat capable of temperature control to within 5°C to 100°C is required. A concentric ring or other cover is required to support Kuderna-Danish concentrators.

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

**NR 149.27 Suspension or revocation of certification.** (1) A laboratory may have its certification suspended or revoked for a particular analyte or the entire test category for any of the following reasons:

(a) Failure to analyze a reference sample and follow-up reference sample within the established acceptance limits.

(b) Failure of a certified laboratory to notify the department within 30 days of major changes in personnel, equipment, or laboratory location which may impair analytical capability. A major change in personnel is defined as a situation in which a trained and experienced analyst is no longer available to analyze a particular parameter for which certification has been granted.

(c) Failure of the laboratory to comply with this subchapter based upon an on-site evaluation and failure to correct identified deviations within 30 days of written notification by the department. This includes continued use of unapproved methods and equipment.

(d) Submission of a reference sample to another laboratory for analysis and reporting the data as its own.

(e) Falsification of data.

(2) PROCEDURES FOR SUSPENSION OR REVOCATION AND REAPPLICATION. Procedures in s. NR 149.42 (3) and (4) apply to suspensions, revocations, and reapplication under this section.

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

## Subchapter IV - Administrative Procedures

**NR 149.41 Laboratory evaluations.** (1) The department may conduct an on-site evaluation of each laboratory, subsidiary laboratory, or branch 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.

(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.

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

**NR 149.42 Suspension or revocation of certification or registration.** (1) SUSPENSION OR REVOCATION OF CERTIFICATION. A laboratory's certification is valid until it expires, is suspended or revoked. Causes for suspension or revocation are:

(a) Failure to meet the acceptance limits on the third reference sample or failure to analyze the reference samples within the time limit specified in s. NR 149.13 (8) or (9). Suspension or revocation shall only be for the analyte, test category, or categories in which inability to meet acceptance limits on reference samples or failure to analyze reference samples

has been demonstrated. For test categories 1, 3, 4, 5, 9, 11, 12, 13, 14, 15, 17, and 20, suspension or revocation shall be for the entire test category. For all other test categories, suspension or revocation shall be by analyte within the test category.

(b) Material and consistent failure to implement or comply with a quality control program as specified under s. NR 149.14. Suspension or revocation may be for the analyte, test category, or categories in which failure to implement or comply with a quality control program has been demonstrated.

(c) Falsification of analytical results or any other information submitted by the laboratory or by another party to the department.

(d) Failure to pay annual fee.

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

(f) Material and consistent failure to allow the department or its representative to inspect the laboratory.

(2) SUSPENSION OR REVOCATION OF REGISTRATION. A laboratory's registration is valid until it expires, is suspended or revoked. If the laboratory has materially and consistently failed to comply with the quality control procedures specified in s. NR 149.14, the laboratory's registration may be suspended or revoked by analyte or by test category or categories.

(3) PROCEDURE FOR SUSPENSION OR REVOCATION OF CERTIFICATION OR REGISTRATION. (a) An order suspending or revoking the certification or registration shall be mailed to the laboratory and shall state the reasons for suspension or revocation. The order shall include a timetable for correcting the deficiencies that led to the suspension or revocation, if appropriate.

(b) An order suspending or revoking a certification or registration shall take effect on the thirtieth day after the order is mailed, unless the certified or registered laboratory submits a request for a hearing to the department within 30 days. The request for hearing shall specify the findings or conclusions, or both, which the laboratory disputes. If a request is submitted, the suspension or revocation is 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.

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

(4) REAPPLICATION. A laboratory which has had its certification or registration suspended or revoked may reapply for certification or registration if the suspension or revocation has expired, the requirements of s. NR 149.07 are met and the deficiencies that led to the suspension or revocation have been corrected in accordance with the timetable contained in the order, if applicable. The department shall respond to the reapplica-

tion within 20 business days of receipt of the completed application as specified by s. NR 149.07.

(5) SUBSIDIARY OR BRANCH LABORATORIES. Suspension or revocation for certification or registration shall be for all subsidiary or branch laboratories which are under one certification or registration.

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

**NR 149.43 Reciprocity.** (1) The department may recognize the certification, registration, licensure or approval of a laboratory by a private organization, 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 may not recognize the certification, registration, licensure or approval of a laboratory by a private organization, another state or an agency of the federal government unless that private organization, state or federal agency recognizes laboratories certified under this chapter. Any laboratory which has such a certification, registration, licensure or approval may apply to the department to have the same recognized under this chapter. The department shall periodically publish a list of those private organizations, other states, and agencies of the federal government whose certifications, approvals or registrations it accepts.

(2) The department shall negotiate with and attempt to enter into accepable agreements with federal agencies, agencies of other states and private agencies for the purpose of reciprocal recognition of laboratory certification and registration under this chapter.

(3) 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.

**NR 149.44 Discretionary acceptance and subcontracted work.** (1) DISCRETIONARY ACCEPTANCE. The department may accept 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. 144.95 (5) (d), Stats., if it is necessary to verify the results of a test submitted under this section.

(2) SUBCONTRACTED WORK. The quality control and record requirements for samples sent to a laboratory with which a certified or registered laboratory subcontracts its work shall be in accordance with ss. NR 149.06 and 149.14. The quality control data for those samples sent to a subcontract laboratory shall be available to the department upon request.

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

**NR 149.45 Variances.** (1) GENERAL. The department may, with the advice of the council, approve variances from nonstatutory 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.