CR 92-16

WISCONSIN DEPT. OF NATURAL RESOURCES

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

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STATE OF WISCONSIN)
) ss
DEPARTMENT OF NATURAL RESOURCES)

TO ALL TO WHOM THESE PRESENTS SHALL COME, GREETINGS:

I, Bruce B. Braun, Deputy Secretary of the Department of Natural Resources and custodian of the official records of said Department, do hereby certify that the annexed copy of Natural Resources Board Order No. TS-45-91 was duly approved and adopted by this Department on June 25, 1992. I further certify that said copy has been compared by me with the original on file in this Department and that the same is a true copy thereof, and of the whole of such original.

RECEIVED
OCT 1 1992
9:30 am
Revisor of Statutes
Bureau

IN TESTIMONY WHEREOF, I have hereunto set my hand and affixed the official seal of the Department at the Natural Resources Building in the City of Madison, this 21st day of September, 1992

Bruce B. Braun

Bruce B. Braun, Deputy Secretary

(SEAL)

Publ 12-1-92
Publ 1-1-93
Publ 1-1-93

ORDER OF THE STATE OF WISCONSIN NATURAL RESOURCES BOARD
REPEALING, RENUMBERING, AMENDING, REPEALING & RECREATING AND CREATING RULES

IN THE MATTER of repealing subch. I title, ss. NR 149.03(11), 149.03 (23), subch. II title, 149.14(2), 149.14(3) Note, 149.14(3)(i), 149.14(4), subch. III title, 149.23, 149.24, 149.25, 149.26, 149.27, 149.28, subch. IV title 149.44(2), 219.03(3) & (4), and 219.06; renumbering s. NR 219.07; amending ss. NR 149.02, 149.03(5)(a) to (e), 149.03(5)(g) to (i), 149.03(5)(k), 149.03(5) Note,149.03(6), 149.03(13) to (17), 149.03 (22) and (29), 149.04(2), 149.04 Table 1, 149.05(1)(b) and (c), 149.05(2) to (3) and (5), 149.06(1) to (1)(c), 149.06(2)(b)6, 149.06(1) Note, 149.07(1)(b)3, 149.07(1)(c), 149.07(1)(e) to (f), 149.07(4), 149.11(2) Note, 149.11(3), 149.11(5), 149.13(1), 149.13(3) to (5), 149.13(7), 149.13(10) to (11)(b), 149.14(1), 149.14(3)(a) to (5)(f)3, 149.14(3)(g) to (h), 149.14(3)(j)1 to (j)3, 149.14(3)(k), 149.14(5), 149.41(1), 149.44(1), and 219.04; repealing and recreate ss. NR 149.03 (5)(l), 149.03(18), 149.05(1)(g), 149.06(4), 149.07(2) to (3), 149.07(5),149.11(1), 149.11(4), 149.12, 149.13(2), 149.13(6), 149.21, 149.22, 149.42, 157.20, 157.21, 219.03(1), 219.05, ch. NR 219 tables A, B, C, D, E & F, and 347.06(5) & (6); and creating s. NR 149.01 Note, 149.03(5)(m), 149.03(8h) and (8q), 149.03(21m), 149.03(22m), 149.03(26g), 149.03(26r), 149.03(29m), 149.03(33), 149.04(3), 149.05(1)(h) and (i), 149.06(1)(e) to (g), 149.06(5), 149.07(1m), 149.11(6) to (7), 149.14(3)(f)4, 149.14(3)(j)4, 149.14(6), 149.41(3), and 149.46 of the Wisconsin Administrative Code pertaining to laboratory certification and registration, preservation procedures and laboratory procedures.

TS-45-91

Analysis Prepared by Department of Natural Resources

Statutory authority: ss. 144.79(8), 144.95, 147.08(1), and 227.11, Stats.

Statutes interpreted: ss. 144.79(8), 144.95, and 147.08(1), Stats.,

The effect of amending ch. NR 149 is to incorporate improvements relating to the enforcement of current rule requirements, restructure the test categories, add test categories for the certification and registration of laboratories testing effluent toxicity and petroleum hydrocarbons, address short comings in the current rule, clarify the rule, and simplify the rule.

The effect of repealing and recreating tables A, B, C, D, E & F will be to delete out of date analytical references, cite more current analytical references, add analytical methodology not preoviously cited, and precribe sample preservation procedures. The recreated tables reflect the federal requirements which were revised in the June 15, 1990, August 15, 1990, and October 8, 1991, federal registers.

The effect of repealing and recreating ss. NR 157.20 and NR 157.21 is to update the references cited for analytical methodology and clarify the request for alternate test procedure approval.

The effect of amending ss. NR 347.06 (5) and (6), is to clarify the requirements for sample preservation, analytical procedures, and use of a certified or registered laboratory when testing sediment samples.

SECTION 1. Subchapter I (title) of chapter NR 149 is repealed.

SECTION 2. NR 149.01 Note is created to read:

NR 149.01 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.

SECTION 3. NR 149.02 is amended to read:

NR 149.02 APPLICABILITY. (1) ~~Except as provided in subs. (2) and (3), the provisions of subchs. I, II & IV are applicable~~ This chapter applies to laboratories applying for ~~certifications~~ certification or ~~registrations~~ registration 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 & IV are applicable~~ Subsection 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 ~~ss. NR 109.12, 109.13, 109.14, 109.21, and 109.22~~ ch. NR 109.

(3) This chapter ~~is~~ does not ~~applicable~~ 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.

Note: Administrative codes requiring analyses to be done by a certified or registered laboratory are: chs. NR 109, 110, 113, 123, 131, 132, 140, 145, 150, 157, 158, ~~180, 181,~~ 182, 210, 211, 212, 219 ~~and~~, 347, 508, 550, 605 and 630.

SECTION 4. NR 149.03 (5)(a) to (e), (g) to (i), and (k) is amended to read:

NR149.05 (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~~ 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, ~~Appendixcies~~ Appendicies 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, ~~July, 1982~~ November, 1986, including ~~update number 1, 1984~~ December 1987 and November 1990 updates.

(d) "Standard Methods for the Examination of Water and Wastewater", ~~16th~~ 17th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, ~~1985~~ 1989.

(e) "~~1984~~ 1991 Annual Book of ASTM Standards, Section 11.01 ~~and~~, 11.02, and 11.04, Water and Environmental Technology", American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

(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~~ 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 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 ~~St. Claire~~ Martin Luther King Drive, Cincinnati, Ohio 45268, March, 1979.

(k) "Official Methods of Analysis of the Association of Official Analytical Chemists, ~~14th~~ 15th edition, Association of Official Analytical Chemist, P.O. Box 540, Washington, D.C. 20044, ~~1985~~ 1990.

SECTION 5. NR 149.03 (5)(l) are repealed and recreated to read:
NR 149.03(5) (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.

SECTION 6. NR 149.03 (5)(m) is created to read:
NR 149.03 (5) (m) "Methods for the Determination of Metals in Environmental Samples", EPA/600/4-91/010, Office of Research and Development, June 1991.

SECTION 7. NR 149.03 (5) Note is amended to read:
NR 149.03 (5) 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. ~~(a),~~ (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), (g), ~~and~~ (l), and (m) may be obtained from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

SECTION 8. NR 149.03 (6) is ameded to read:
NR149.03 (6) "Blind standard" means a standard or a sample with a ~~known amount~~ 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.

SECTION 9. NR 149.03 (8h) and (8q) are created to read:
NR 149.03 (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.

SECTION 10. NR 149.03 (11) is repealed.

SECTION 11. NR 149.03 (13) to (17) are amended to read:
NR 149.03 (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 ~~equipment~~ system.

(14) "Laboratory" means a facility~~, subsidiary facilities, or branch facilities under common ownership and control~~ which performs tests in connection with a program which requires data from a certified or registered.

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

(15) "Limit of detection" means the lowest concentration level that can be determined to be significantly different from a blank~~ for that analytical test method and sample matrix~~.

(16) "Limit of quantitation" means the ~~concentration of an analyte at which one can state with a stated degree of confidence for that analytical test method and sample matrix that an analyte is present at a specific concentration in the sample tested~~ level above which quantitative results may be obtained with a specified degree of confidence.

~~Note: The above "limit of detection" and "limit of quantitation" definitions are intended to be equivalent to the definitions in ch. NR 140 for "limit of detection" and "limit of quantitation."~~

(17) "Method of standard addition" means an analytical technique used to quantify samples whose matrices differ significantly from those of the known standards which is accomplished by analyzing the sample ~~mixed~~ and mixtures of sample with at least 3 known standards~~ and a blank~~, plotting the response versus added concentration and extrapolating the plot to determine the original concentration of the analyte in the sample.

SECTION 12. NR 149.03 (18) is repealed and recreated to read:
NR 149.03 (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 blank may be advisable. The matrix blank may not contain the analyte above the level of detection.

SECTION 13. NR 149.03 (21m) is created to read:
NR 149.03 (21m) "Qualify" means to place a written statement accompanying the test results which identifies anomalies encountered in generating the data.

SECTION 14. NR 149.03 (22) and (29) are amended to read:
NR 149.03 (22) "Quality control limit" means the calculated acceptance limits determined using a procedure from an authoritative source for ~~duplicate~~ replicate and spiked sample analysis ~~that have been determined using a procedure from an authoritative source~~ or other quality control checks.

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

SECTION 15. NR 149.03 (22m) is created to read:
NR 149.03 (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.

SECTION 16. NR 149.03 (23) is repealed.

SECTION 17. NR 149.03 (26g), (26r), (29m) and (33) are created to read:

NR 149.03 (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.

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

(35) "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.

SECTION 18. NR 149.04 (2) is amended to read:
NR 149.04 (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~~ s. NR 149.21.

SECTION 19. NR 149.04 (3) is created to read:
NR 149.04 (3) The effluent toxicity test category has specific requirements which are described in s. NR 149.22.

SECTION 20. NR 149.04 Table 1 is amended to read:

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, Oil and Grease.
5.	General I	~~Chloride~~ Hardness	Alkalinity/Acidity, ~~Chloride,~~ Bromide, Chlorophyll a, Color, Hardness, ~~Sulfate~~ 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~~	~~Bromide, Color, Odor, Oil and Grease, Specific Conductance, Sulfide, Sulfite, Surfactants, Turbidity~~
~~8.~~7.	General ~~IV~~ III	No reference sample	~~Corrosivity,~~ EP Toxicity, Ignitability, Reactivity, Waste Fingerprinting Analyses, Total Organic Carbon, Total Organic Halide, Toxicity Characteristic Leaching Procedure.

TABLE 1
Test Categories

No. Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
~~9.~~8. Metals I	~~Copper and Cadmium~~ 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, Silicon,~~ Selenium, Silver, Sodium, Strontium, Thallium, Tin, ~~Titanium, Tungsten,~~ Vanadium, and Zinc~~, and Zirconium~~.
~~10.~~9. 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.
~~11.~~10. Organics; Purgeable by Gas Chromatography or Gas Chromatography/ Mass ~~Spectrometer~~ Spectrometry	~~Trichloroethene and Benzene~~ Representative purgeable analytes.	Purgeable Halocarbons, Purgeable Aromatics, Acrolein, Acrylonitrile.

Table 1 Continued

No. Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
~~12.~~11. Organics; Base/Neutral Extractables by Gas Chromatography or Gas Chromatography/Mass ~~Spectrometer~~ Spectrometry	~~P,P'-DDT and Dieldrin~~ Representative analytes within each analyte group for which certification or registration is desired. The following groups are included: Base Neutral Pesticides, Phthalate Esters, Nitrosamines, Polynuclear Aromatics, and Haloethers.	Benzidine, 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.~~12. ~~Organic~~ Organics; Acid Extractables by Gas Chromatography or Gas Chromatography/Mass ~~Spectrometer~~ Spectrometry	~~Pentachlorophenol~~ Representative Acid Extractable Organic analytes.	Phenolic Compounds.
~~14.~~13. Organics; Extractables by Liquid Chromatography	~~Naphthalene~~ Representative Polynuclear Aromatic Hydrocarbons or Pesticides analyzable by liquid chromatography.	Benzidines, Polynuclear Aromatic Hydrocarbons, Pesticides subject to Liquid Chromatography (e.g., carbofuran, oxamyl, and methomyl).
~~15.~~14. Organics; Acid Extractable Pesticides	~~2,4-D~~ Representative Acid Extractable Pesticides.	2,4-D, 2,4,5-T, Picloram, Chloramben, and other acid extractable pesticides.

Table 1 Continued

No.	Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
~~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 ability to perform the analysis. See s. NR 149.13(11).~~	~~Aldicarb, Ethylene Dibromide, and Glyphosate, and other pesticides.~~
15.	Organics; Petroleum Hydrocarbons	Gasoline Range Organics (GRO), Diesel Range Organics (DRO), Total Recoverable Petroleum Hydrocarbons (TRPH), Petroleum Volatile Organic Carbon (PVOC).	Gasoline Range Organics, Diesel Range Organics, Petroleum Volatile Organic Carbon (PVOC), and Total Recoverable Petroleum Hydrocarbons (TRPH).
~~17.~~16.	~~Organic Polychlorinated Biphenyls~~ Organics; Organochlorine Compounds	~~PCB (Common Aroclor)~~ Representative analytes within the Aroclors and Organochlorine pesticides groups for which certification or registration is desired.	Polychlorinated Biphenyls and Organochlorine Pesticides.

Table 1 Continued

No. Test Category	Key Analyte	Analytes In Test Category (Includes all forms of the given analytes)
~~18.~~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.
~~19.~~18. Safe Drinking Water	Each analyte or analyte group for which certification is desired.	Arsenic, Asbestos, Barium, Cadmium, Chromium, Copper, Fluoride, Lead, Mercury, Nitrate as Nitrogen, Nitrite as Nitrogen, Selenium, ~~Silver,~~ Alachlor, Atrazine, Carbofuran, Chlordane, Dibromochloropropane, Endrin, Ethylene Dibromide, Heptachlor, Heptachlor Epoxide, Lindane, Methoxychlor, Polychlorinated Biphenyls, Toxaphene, 2,4-D, 2,4,5, - TP, Total Trihalomethanes, Benzene, Vinyl Chloride, Carbon Tetrachloride, 1,2-~~d~~Dichloroethane, ~~t~~Trichloroethylene, 1,1-~~d~~Dichloroethylene, 1,1,1-~~t~~Trichloroethane, para-~~d~~Dichlorobenze, 1,2-Dichloropropane, Ethylbenzene, Chlorobenzene, o-Dichlorobenzene, Styrene, Tetrachloroethylene, Toluene, Trans-1,2-Dichloroethylene, Xylenes.

Table 1 Continued

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

~~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.~~

SECTION 21. NR 149.05 (1)(b) and (c) are amended to read:

NR 149.05 (1) (b) Annual fee for each test category except the safe drinking water and the effluent toxicity test ~~category~~ categories - $25

(c) Annual fee for safe drinking water test category - $300. If a laboratory only wishes certification for nitrate-nitrogen, the fee is $50.

SECTION 22. NR 149.05 (1)(g) is repealed and recreated to read:
NR 149.05 (1) (g) On-site evaluation fee for enforcement follow-up evaluation - Actual cost of evaluation.

SECTION 23. NR 149.05 (1)(h) and (i) are created to read:
NR 149.05(1) (h) Annual fee for effluent toxicity test category - $300

(i) Annual fee for laboratories accepted under reciprocity agreements - $150

SECTION 24. Ammend s. NR 149.05 (2), (3) and (5).

(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, ~~reference samples,~~ laboratory evaluations, discretionary acceptance of data, reciprocity, and collection of fees.

(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~~ the midpoint of the certification or registration period.

SECTION 25. NR 149.06 (1)(intro.), (a) to (1)(c) are amended to read:
NR 149.06 (1)(intro.) ~~The following records~~ 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. Records to be retained include but are not limited to records of the following:

(a) ~~A record of samples~~ 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 data for spikes, ~~duplicates~~ replicates, ~~reagent~~ 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 ~~each parameter~~ spikes and replicates.

SECTION 26. NR 149.06 (1)(e) through (g) are created to read:
NR 149.06 (1) (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.

SECTION 27. NR 149.06 (1) Note is amended to read:
NR 149.06 (1) Note: Chapter NR 109, safe drinking water program ~~contained in subch. III,~~ requires that the actual chemical sample results be retained for 10 years by the agency responsible for the drinking water supply.

SECTION 28. NR 149.06 (2)(b)6 is amended to read:
NR 149.06 (2)(b) 6. Location, date, collector's name and time of sampling.

SECTION 29. NR 149.06 (4) is repealed and recreated to read:
NR 149.06 (4) Upon the department's request, a certified or registered laboratory shall submit to the department records, under sub. (1), from any subcontracted laboratories.

SECTION 30. NR 149.06 (5) is created to read:
NR 149.06 (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.

SECTION 31. NR 149.07 (1)(b)3, (c), and (e) to (f) are amended to read:
NR 149.07 (1)(b) 3. ~~Reference~~ 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 ~~or NR 149.21~~.

(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.

SECTION 32. NR 149.07 (1m) is created to read:

NR 149.07 (1m) 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.

SECTION 33. NR 149.07 (2) to (3) are repealed and recreated to read:
NR 149.07 (2) 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 reciept 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.

(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. 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.

SECTION 34. NR 149.07 (4) is amended to read:
NR 149.07 (4) 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 ~~July 1~~ 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 ~~analysis and results required under s. NR 149.13(4) shall be completed~~ results prior to renewal.

SECTION 35. NR 149.07 (5) is repealed.

SECTION 36. Subchapter II (title) of ch. NR 149 is repealed.

SECTION 37. NR 149.11 (1) is repealed and recreated to read:
NR 149.11 (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 authoritive 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 ~~law~~ regulations are in chs. NR 109, 219, ~~181,~~ 508 and 605. Those required by federal ~~law~~ regulations are in 40 CFR 136, 141 and 261.

SECTION 38. NR 149.11 (2) Note and (3) are amended to read:
NR 149.11 (2) Note: Sample collection methods required by state ~~law~~ regulations are in chs. NR 218~~,~~ and 140~~ and 181~~.

(3) Sample preservation procedures and holding times required by state and federal ~~laws~~ regulations shall be followed. If the sample preservation procedures and holding times are not required by state or federal ~~laws~~ regulations, 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~~ improperly 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 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.

SECTION 39. NR 149.11 (4) is repealed.

SECTION 40. NR 149.11 (5) is amended to read:
NR 149.11 (5) If requested, the ~~laboratory shall determine the~~ limit of quantitation and limit of detection shall be determined in accordance with ~~s. NR 149.03(5)(b) and (j)~~ a method specified by the department.

SECTION 41. NR 149.11 (6) to (7) are created to read:
NR 149.11 (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.

SECTION 42. NR 149.12 are repealed and recreated to read:

NR 149.12 ALTERNATE METHODOLOGY. Laboratories may use alternate methodologies other than those prescribed in ch. NR 149 if EPA has granted an approval for their use. The laboratory shall provide 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 state ~~law~~ regulations in ch. NR 219 and by federal ~~law~~ regulations in 40 CFR 136, 141, 260 and 403.

SECTION 43. NR 149.13 (1) is amended to read:

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.

SECTION 44. NR 149.13 (2) is repealed and recreated to read:

NR 149.13 (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.

SECTION 45. NR 149.13 (3) to (5) are amended to read:

NR 149.13 (3) ~~The department may provide each certified laboratory with~~ 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~~, or the laboratory may procure its own reference samples~~. ~~For a~~ A registered laboratory shall successfully analyze and report results of 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~~ Wisconsin state laboratory of hygiene 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~~ department shall maintain a list of approved reference samples sources~~:~~.

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

~~(b) U.S. 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 include all of the following ~~are~~:

~~1.~~(a) 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~~ those used by the Wisconsin state laboratory of hygiene.

~~2.~~(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.

~~3.~~(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 ~~provided by the department or a source~~ from an approved ~~under sub. (3)(d)~~

source shall be analyzed and reported to the ~~department between February 1 and March 31 of each year~~ 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). ~~Additional reference sample results may be submitted after March 31.~~

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

SECTION 46. NR 149.13 (6) is repealed and recreated to read:
NR 149.13 (6) The Wisconsin State Laboratory of Hygiene shall use standard statistical methods, with the concurrence of the council, to determine the acceptance limits.

SECTION 47. NR 149.13 (7) and (10), (11)(intro.) and (b) are amended to read:
NR 149.13 (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~~.

(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~~ 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)(intro.) For test ~~categories 16 and 18~~ category 17, no reference sample is required. The laboratory shall demonstrate, upon application for certification or registration, acceptable precision and percent recovery based on ~~duplicate~~ replicate analysis and spiked sample analysis. The following information shall be submitted:

(b) Results of 15 samples analyzed in ~~duplicate~~ replicate using the above submitted methodology. Samples chosen for ~~duplicate~~ replicate 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.

SECTION 48. NR 149.14 (1) is amended to read:
NR 149.14 (1) Each laboratory shall maintain a quality control program. The quality control program shall include a written quality assurance plan. The quality control ~~program~~ data shall be documented and such documents shall be available, upon request, to the department.

SECTION 49. NR 149.14 (2) is repealed.

SECTION 50. NR 149.14 (3)(a) to (f)3 are amended to read:
NR 149.14 (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 ~~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). 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, ~~9~~ 8, ~~10~~ 9 and for total organic carbon, total organic halide, ~~chloride,~~ and hardness, ~~and sulfate,~~ 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, ~~and 18,~~ 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, ~~corrosivity, EP toxicity, ignitability, reactivity, gravimetric tests, titratimetric tests,~~ color, odor and analysis under test ~~categories~~ category 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 or those established by the provider.

5. For test category ~~20~~ 19 the pre-established limit shall be appropriate for the test.

(d) At least one ~~reagent~~ method blank shall be analyzed on each analysis day, for those tests for which ~~reagent~~ method blanks are appropriate. For certain tests, a nonreacted sample may be used as a blank. There is no requirement to run a blank for solids testing.

~~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~~ replicate sample shall be run after the analysis of 10 samples for each matrix type, unless the methodology specifies otherwise. ~~For those methodologies which require that the sample bottle be extracted, duplicate samples shall be taken in the field to insure representative samples.~~ No replicate samples are needed for oil and grease.

(f) Spiked samples shall be analyzed for each matrix type 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 as cited in the approved method or authoritative source. If no frequency is given, then the minimum frequency shall be:

1. ~~As required in the authoritative sources~~ After the analysis of 10 samples, for test categories ~~11~~ 10 to ~~18~~ 17, ~~20~~ 19, 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, 5, 6, 8, and 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, chlorophyll a, 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.

SECTION 51. NR 149.14 (3)(f)4 is created to read:
NR 149.14 (3)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.

SECTION 52. NR 149.14 (3) Note is repealed.

SECTION 53. NR 149.14 (3)(g) to (h) are amended to read:
NR 149.14 (3)(g) Quality control limits for ~~duplicate~~ 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 ~~30~~ 20 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, spiked samples, or ~~duplicates~~ replicates 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,~~ When the attempted corrective action does not solve the problem the laboratory shall reanalyze the affected 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.

SECTION 54. NR 149.14 (3)(i) is repealed.

SECTION 55. NR 149.14 (3)(j) is amended to read:
NR 149.14 (j) ~~A blind standard~~ Blind standards shall be analyzed~~, if available for that analyte, every 4 months for~~ 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 ~~10~~ 9, ~~16~~ 15, and ~~20, if the analyte was analyzed during the previous 4-month period~~ 19.

2. ~~A blind standard shall be analyzed every 4 months~~ Analysis shall be conducted for one analyte in each test category in test categories ~~11~~ 10 to ~~15~~ 14, ~~17~~ 16, and ~~18, if an analyte within those test categories was analyzed during the previous 4-month period~~ 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.

SECTION 56. NR 149.14 (3)(j)4 is created to read:
NR 149.14 (3)(j)4. The blind standard shall be analyzed at least 3 months and no longer than 5 months after the previous blind standard.

SECTION 57. NR 149.14(3)(k) is amended to read:
NR 149.14(3)(k) Where ~~duplicate~~ 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.

SECTION 58. NR 149.14 (4) is repealed.

SECTION 59. NR 149.14 (5)is amended to read:
(5) If it has been determined that an organic analyte is present in ~~a~~ an unfamiliar sample, the laboratory shall ~~inform the data user if the results have been confirmed by a second analysis with a different methodology~~ confirm the results, unless the analysis is done by mass spectrometry.

SECTION 60. NR. 149.14 (6) is created to read:
(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.

SECTION 61. Subchapter III (title) of ch. NR 149 is repealed.

SECTION 62. NR 149.21 is repealed and recreated to read:
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 109.
(1) FLUORIDE. Fluoride analyses required under s. NR 109.705 need not be performed by a certified laboratory.
(2) FREE CHLORINE RESIDUAL. Free chlorine residual and total chlorine residual analyses required under s. NR 109.705 need not to be done by a certified laboratory.
(3) ANALYSIS FOR pH. Analyses for pH required under s. NR 109.14 need not be done by a certified laboratory.
(4) TURBIDITY. Turbidity analyses as required under s. NR 109.41 need not be done by a certified laboratory.
Note: 40 CFR 141.28 excludes turbidity, free chlorine residual and pH from certification.
(5) REQUIREMENTS FOR INORGANIC CHEMICALS. To receive certification to conduct analyses for asbestos, barium, cadmium, chromium, copper, fluoride, lead, mercury, nitrate, nitrite and selenium the laboratory shall:
(a) Analyze reference samples for these substances, provided by EPA or another approved source, and achieve quantitative results on the analyses that are within the following acceptance limits:

Contaminant	Acceptance Limit
Asbestos	2 standard deviations based on study statistics.

Barium	±15% at ≥0.15 mg/L
Cadmium	±20% at ≥0.002 mg/L
Chromium	±15% at ≥0.01 mg/L
Copper	±10% at ≥0.050 mg/L
Fluoride	±10% at 1 to 10 mg/L
Lead	±30% at ≥0.005 mg/L
Mercury	±30% at ≥0.0005 mg/L
Nitrate	±10% at ≥0.4 mg/L
Nitrite	±15% at ≥0.4 mg/L
Selenium	±20% at ≥0.01 mg/L

(b) Achieve a limit of detection of 0.001 mg/L for lead and 0.001 mg/L for copper unless atomic absorption direct aspiration is used and then the limit of detection for copper shall be 0.020 mg/L.

(6) REQUIREMENTS FOR VOLATILE ORGANIC COMPOUNDS. To receive certification to conduct analyses for benzene, vinyl chloride, carbon tetrachloride, 1,2-dichloroethane, trichloroethylene, 1,1-dichloroethylene, 1,1,1-trichloroethane, paradichlorobenzene, 1,2-dichloropropane, ethylbenzene, chlorobenzene, o-dichlorobenzene, styrene, tetrachloroethylene, toluene, trans-1,2-dichloroethylene, and xylenes the laboratory shall:

(a) Analyze reference samples which include these substances provided by EPA or another approved source; and

(b) Achieve quantitative results on the analyses performed under subd. 1. that are within ± 20% of the actual amount of the substances in the reference sample when the actual amount is greater than or equal to 0.010 mg/L; and

(c) Achieve quantitative results on the analyses performed under subd. 1. that are within ± 40% of the actual amount of the substances in the reference sample when the actual amount is less than 0.010 mg/L; and

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

(e) Except for vinyl chloride, achieve a limit of detection of 0.0005 mg/L, according to the procedures in 40 CFR 136 Appendix B. To receive certification for vinyl chloride, the laboratory shall achieve a limit of detection of 0.0003 mg/L, according to the procedures in 40 CFR 136 Appendix B.

(7) REQUIREMENTS FOR OTHER ORGANIC COMPOUNDS To receive certification to conduct analyses for the following contaminants, the laboratory shall analyze reference samples provided by EPA or another approved source, and achieve quantitative results on the analyses that are within the following acceptance limits:

Contaminant	Acceptance Limit
DBCP	±40%
EDB	±40%
Alachlor	±45%
Atrazine	±45%
Carbofuran	±45%
Chlordane	±45%
Heptachlor	±45%
Lindane	±45%
Methoxychlor	±45%
PCBs(as Decachlorobiphenyl)	0-200%
Toxaphene	±45%
Aldicarb	2 standard deviations based on study statistics
Aldicarb sulfoxide	2 standard deviations based on study statistics
Aldicarb sulfone	2 standard deviations based on study statistics
Pentachlorophenol	±50%
2,4-D	±50%
2,4,5-TP	±50%

(8) 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.

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.

SECTION 63. NR 149.22 is repealed and recreated to read:

NR 149.22 REQUIREMENTS FOR EFFLUENT TOXICITY CERTIFICATION AND REGISTRATION. This section applies to those laboratories certified or registered under test category 20. The quality control requirements given in s. NR 149.14 do not apply to effluent toxicity testing. The required quality control procedures along with the criteria and procedures for effluent toxicity testing are given in the approved methods and the "Guidance Manual for the Certification and Registration of Laboratories Conducting Effluent Toxicity Testing", Wisconsin Department of Natural Resources, May 1992.

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, Office of Technical Services, P.O. Box 7921, Madison, WI 53707.

Note: The approved methods are cited in ch. NR 219.

SECTION 64. Subchapter IV (title) of ch. NR 149 is repealed.

SECTION 65. 149.23, 149.24, 149.25, 149.26, 149.27 and 149.28 are repealed.

SECTION 66. NR 149.41(1) is amended to read:

(1) The department ~~may~~ shall 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. 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.

SECTION 67. NR 149.41 (3) is created to read:
NR 149.41 (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.

SECTION 68. NR 149.42 is repealed and recreated to read:

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.

(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.

(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 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.

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. Any violation of this chapter may be referred to the attorney general's office for enforcement under ss. 144.98 and 144.99, Stats.

SECTION 69. NR 149.44 (title) and (1) are ammended to read:

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. This section does not apply to monitoring required under ch. NR 109, where a certified laboratory is required.

SECTION 70. NR 149.44 (2) is repealed.

SECTION 71. NR 149.46 is created to read:

NR 149.46 PROCEDURES FOR REVISING CERTIFICATION OR REGISTRATION AS A RESULT OF THE 1992 AMENDMENTS. (1) LABORATORIES HOLDING VALID CERTIFICATION OR REGISTRATION PRIOR TO THE EFFECTIVE DATE OF THE AMENDMENTS TO s. NR 149.04. The department shall certify or register laboratories for the test categories containing the same analytes for which the previous certification or registration was valid. The laboratory shall meet the requirements of s. NR 149.07(1)(b)1, (c), (d) and (e). Prior to the effective date of s. NR 149.04, the department shall provide and the laboratories shall complete and submit a status update form to facilitate the conversion of the test categories and demonstrate that these requirements have been met.

(a) Laboratories holding valid certification or registration for oil and grease under the general III test category but not the physical test category shall become certified or registered in category 19, any single analyte.

(b) Laboratories holding valid certification or registration for purgable organics or purgable aromatics may obtain certification or registration for organics; petroleum hydrocarbons by meeting the requirements of sub. (1)(intro.).

(c) The department may not adjust fees for the conversion to the amended test category structure in Table 1 and no fee may be assessed for reissuing certificates as a result of this conversion.

(2) CERTIFICATION OR REGISTRATION FOR ADDITIONAL TEST CATEGORIES. If the laboratory wishes to become certified or registered in additional test categories, the laboratory shall comply with provisions of s. NR 149.07. The laboratory may apply for the additional test categories on the status update form.

(3) LABORATORIES RECOGNIZED THROUGH RECIPROCITY WITH ANOTHER STATE. The provisions of subs. (1) and (2), with the exception of par. (1)(b), shall apply to laboratories recognized under reciprocity.

SECTION 72. NR 157.20 and 157.21 are repealed and recreated to read:

NR 157.20 TESTING METHODS FOR PCBS AND PRODUCTS CONTAINING PCBS. (1) For transformer fluids, waste oils, insulating liquids, and other non-polar liquids containing PCBs the procedures and gas chromatographic analysis used

shall be as defined in "Standard Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography", ASTM standard D 4059-86, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103; or "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils", EPA-600/4-81-045, U.S. EPA Monitoring and Support Laboratory, Cincinnati, OH.

(2) For paper and paperboard containing PCBs extraction procedures and gas chromatographic analysis used shall be as defined in the "Official Methods of Analysis of the AOAC", page 285, 15th edition.

Note: This publication maybe obtained from the Association of Official Analytical Chemists, 2200 Wilson Blvd., Suite 400-CV, Arlington, VA 22201-3301.

(3) For leachate, non-drinking groundwaters, soils, sediments, and sludges containing PCBs not regulated by a Wisconsin pollution discharge elimination system permit, the extraction procedures and gas chromatographic analysis used shall be as defined in the method 8080A found in "Test Methods for Evaluating Solid Waste", SW-846, U.S. EPA, Update I, November 1990, 3rd edition, November 1986.

Note: Available from the Superintendent of Documents, U.S. Government Printing Office, Washington D.C. 20402.

(4) For wastewater and wastewater treatment sludges containing PCBs, testing methods are defined in ch. NR 219.

(5) For public drinking waters containing PCBs, testing methods are defined in ch. NR 109.

Note: Copies of the above publications are available for inspection at the offices of the Department of Natural Resources, the Secretary of State, and Revisor of Statutes.

(6) Laboratory test results submitted to the department under this chapter shall be performed by a laboratory certified or registered under ch. NR 149.

NR 157.21 APPROVAL OF ALTERNATE TEST PROCEDURES. Applications for approval of alternate test procedures for wastewater analysis must be made as directed in s. NR 219.05.

SECTION 73. NR 219.03 (1) is repealed and recreated to read:
NR 219.03 (1)"EPA" means the U.S. environmental protection agency.

SECTION 74. NR 219.03 (3) and (4) are repealed.

SECTION 75. NR 219.04 is amended to read:

NR 219.04 IDENTIFICATION OF TEST PROCEDURES. (1) ANALYTICAL TEST PROCEDURES. Parameters or pollutants, for which analytical methods are approved, are listed together with test procedure descriptions and references in tables A to E. The discharge values for the listed ~~effluent~~ parameters shall be determined by one of the standard analytical test procedures identified in a table under this subsection or by a alternate test procedure established under ss. NR 219,05 and 219.06.

(2) PRESERVATION PROCEDURES. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to E are prescribed in table F. Sludge samples shall be preserved at the time of collection by cooling to 4°C. Any person may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the regional administrator and shall provide sufficient data to assure that the

variance does not adversely affect the integrity of the sample. The regional administrator will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

SECTION 76. NR 219.05 is repealed and recreated to read:

NR 219.05 ALTERNATE TEST PROCEDURES. Approvals of alternate test procedures for nationwide use and specific discharges are granted by EPA. An alternate test procedure may only be used if the procedure has been approved by EPA.

Note: The federal requirements for alternate test procedure approval are given in 40 CFR 136.5.

SECTION 77. NR 219.06 is repealed.

SECTION 78. NR 219.07 is renumbered to NR 219.06 and NR 219.06 (intro.) as renumbered is amended to read:

NR 219.06 LABORATORY CERTIFICATION OR REGISTRATION. Bacteriological analyses of groundwater samples, and all radiological analysis shall be performed by the state laboratory of hygiene or a laboratory certified or approved by the department of health and social services. Other laboratory test results, including effluent toxicity, submitted to the department under this chapter shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

SECTION 79. Tables A through F of ch. NR 219 are repealed and recreated to read:

TABLE A

LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter and Units	Method[1]	EPA	Standard Methods 17th Ed.	USGS
Bacteria:				
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution; or, membrane filter (MF)[2], single step.	p132[3] p124[3]	9221C 9222C	B-0050-85[4]
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or MF, single step[5]	p132[3] p124[3]	9221C 9222D	
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF[2] single step or two step	p114[3] p108[3]	9221B 9222B	B-0025-85[4]
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or, MF[2] with enrichment.	p114[3] p111[3]	9221B 9222B+B.5c	
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF[2], or Plate count	p136[3] p136[3] p143[3]	9230B 9230C	B-0055-85[4]
Enteroviruses:				
6. Enteroviruses in water, plaque forming units per liter.	Absorption, elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 6[6] Ch. 9[6] Ch. 10[6] Ch. 12[6]	9510 B,C,D,E 9510 G 9510 G 9510 G	
7. Enteroviruses in sludge, plaque forming units per liter.	Beef extract elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 7[6] Ch. 9[6] Ch. 10[6] Ch. 12[6]	9510 F 9510 G 9510G 9510G	
Mutagenicity:				
8. Mutagenicity (revertants per liter)	Ames test, test strains TA97, TA98, TA100, and TA102.	Note 7		
Acute and Chronic Toxicity:				
9. Toxicity, acute, fresh water organisms, effluent[10]	Daphnia and Ceriodaphnia, 48-h static mortality.	p 39[8]		
	Fathead minnow, 48-h static mortality, or 48 to 96-h flow-through mortality.	p 41[8]		
10. Toxicity, chronic, fresh water organisms, percent effluent.[10]	Fathead minnow larval survival and growth.	1000.0[9]		
	Fathead minnow embryo-larval survival and teratogenicity.	1001.0[9]		
	Ceriodaphnia survival and reproduction.	1002.0[9]		
	Selenastrum growth.	1003.0[9]		

TABLE A NOTES:

[1] The method used must be specified when results are reported.

[2] A 0.45 μm membrane filter (MF) or other port size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

[3] Bordner, R.H., and J.A. Winter, eds. "Microbiological Methods for Monitoring the Environment, Water and Wastes", United States Environmental Protection Agency, EPA-600/8-78-017, 1978. Available from ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. Martin Luther King Drive, Cincinnati, Ohio 45268.

[4] Britton, L.J., and P.E. Greeson, eds. "1988 Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples", edited by et al., U.S. Geological Survey, Techniques of Water-Resources Investigation (USGS TWRI), Book 5 chapter A4, Laboratory analysis, 1977. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

[6] Berg, G., R.S. Safferman, D.R. Dahling, D. Berman, and C.J. Hurst, 1984. USEPA Manual of Methods for Virology. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-84/013. (Chapter 9 revised January 1987; Chapter 10 revised December 1987; Chapter 12 revised May 1988; Chapter 7 revised September 1989).

[7] Williams, L.R., and J.E. Preston, eds. 1983. Interim Procedures for Conducting the Salmonella/Microsomal Mutagenicity Assay (Ames Test). Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Las Vegas, Nevada. EPA/600/4-85/013.

[8] Peltier, W.H., and C.I. Weber, eds. September 1991. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-90/027.

[9] Weber, C.I., W.H. Peltier, T.J. Norberg-King, W.B. Horning, II, F.A. Kessler, J.R. Menkedick, T.W. Neiheisel, P.A. Lewis, D.J. Klemm, Q.H. Pickering, E.L. Robinson, J.M. Lazorchak, L.J. Wymer, and R.W. Freyberg. 1989. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Surface Waters to Freshwater Organisms, Second Edition, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. (EPA/600/4-89/001).

[10] Compliance monitoring must be performed in accordance with the specifications in "Guidance Manual for the Certification and Registration of Laboratories Conducting Effluent Toxicity Testing", Wisconsin Department of Natural Resources, May 1992. Available from the Departement of Natural Resources Office of Technical Services, P O Box 7921, Madison, WI 53707.

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
1. Acidity, as $CaCO_3$, mg/L, Electrometric end point or phenolphthalein end point	305.1	2310 B(4a)	B1067-88		
2. Alkalinity, as $CaCO_3$, mg/L; Electrometric or colorimetric: Titration to pH 4.5, manual Or automated	310.1 310.2	2320 B	D1067-88	I-1030-85	973.43[5]

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

	Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
3.	Aluminum-Total[6], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration,	202.1	3111 D		I-3051-85	
	AA furnace,	202.2	3113 B			
	Inductively coupled plasma (ICP),	200.7[7]	3120 B			
	Direct current plasma (DCP), or			D4190-88		Note 34
	Colorimetric (Eriochrome cyanine R)		3500-A1D			
4.	Ammonia (as N), mg/L: Manual distillation[5] (at pH 9.5):					
	Followed by	350.2	4500-NH_3 B			973.49[5]
	Nesslerization,	350.2	4500-NH_3 C	D1426-79(A)	I-3520-85	973.46[5]
	Titration,	350.2	4500-NH_3 E			
	Electrode,	350.3	4500-NH_3 F & G	D1426-79(D)		
	Automated phenate, or	350.1	4500-NH_3 H	D1426-79(C)	I-4523.85	
	Automated electrode					Note 9
5.	Antimony - Total[6], ug/L:					
	Digestion[6] followed by:					
	AA direct aspiration,	204.1	3111 B			
	AA furnace, or	204.2	3113 B			
	Inductively coupled plasma	200.7[7]	3120 B			
6.	Arsenic - Total[6], ug/L:					
	Digestion[6] followed by	206.5				
	AA (gaseous hydride),		3114	D2972-84(B)	I-3062.85	
	AA furnace,	206.2	3113-4d			
	Inductively coupled plasma,	200.7[7]	3120 B			
	Or, colorimetric (SDDC)	206.4	3500-As	D2972-84(A)	I-3060-85	
7.	Barium-Total[6], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration,	208.1	3111 D		I-3084-85	
	AA furnace,	208.2	3113 B			
	Inductively coupled plasma, or	200.7[7]	3120 B			
	DCP					Note 34
8.	Beryllium-Total[6], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration,	210.1	3111 D	D3654-88(A)	I-3095-85	
	AA furnace,	210.2	3113 B			
	Inductively coupled plasma,	200.7[7]	3120 B			
	DCP, or			D4190-88		Note 34
	Colorimetric (aluminon)		3500-Be D			
9.	Biochemical oxygen demand (BOD5), mg/L:					
	Winkler (Azide modifications)	405.1	5210		I-1578-78[10]	973.44[5] p. 17[11]
	Or electrode method					
10.	Boron-Total, mg/L:					
	Colorimetric (curcumin),	212.3	4500-B B		I-3112-85	
	Inductively coupled plasma, or	200.7[7]	3120 B			
	DCP			D4190-88		Note 34
11.	Bromide, mg/L: Titrimetric	320.1		D1246-82 (C)(1988)	I-1125-85	p. S44[12]
12.	Cadmium-Total[6], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration,	213.1	3111 B or C	D3557-90(A or B)	I-3135-85 or	974.27[5] p.37[11]

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
				I-3136-85	
AA furnace,	213.2	3113B			
Inductively coupled plasma	200.7[7]	3120B		I-1472-85	
DCP,			D4190-90		Note 34
Voltametry[13], or			D3557-90(C)		
Colorimetric (Dithizone)		3500-Cd D			
13. Calcium-Total[6], mg/L:					
Digestion[6] followed by:					
Atomic absorption,	215.1	3111 B	D511-88(B)	I-3152-85	
Inductively coupled plasma,	200.7[7]	3120 B			
DCP, or					Note 34
EDTA titration	215.2	3500-Ca D	D511-88(A)		
14. Carbonaceous Biochemical oxygen demand ($CBOD_5$), mg/L: with nitrification inhibitor[14]		5210 B			
15. Chemical oxygen demand (COD), mg/L:					
Titrimetric	410.1	5220 B	D1252-88	I-3560 or	973.46[5] p.17[11]
	410.2			I-3562-85	
	410.3				
Automated and manual Spectrophotometric	410.4			I-3561-	Notes 15 or 16
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or		4500-Cl^- B	D512-89(B)	I-1183-85	
(Mercuric nitrate),	325.3	4500-Cl^- C	D512-89(A)	I-1184-85	973.51[5]
Colorimetric (ferricyanide), manual			D512-89(C)	I-1187-85	
or automated	325.1 or	4500-Cl^- E		I-2187-85	
	325.2				
17. Chlorine - Total residual, mg/L:					
amperometric,	330.1	4500-Cl D	D1253-76(A)		
Starch End point direct	330.3	4500-Cl B	D1253-76(B) (1985) Part 18.3		
Back Titration either end point[17],or	330.2	4500-Cl C			
DPD-FAS,	330.4	4500-Cl F			
Spectrophotometric, DPD; or	330.5	4500-Cl G			
Electrode					Note 18
18. Chromium VI dissolved, ug/L: 0.45 micron filtration with:					
Extraction and atomic absorption, or	218.4	3111 A		I-1232-85	
Colorimetric (Diphenylcarbazide)				I-1230-85	307B[19]
19. Chromium-Total[6], mg/L:					
Digestion[6] (optional extraction) followed by:					
AA direct aspiration,	218.1	3111 B	D1687-86(D)	I-3236-85	974.24[5]
AA chelation extraction	218.3	3111 C			
AA furnace,	218.2	3113B			
Inductively coupled plasma,	200.7[7]	3120B			
DCP, or			D4190-88		Note 34
Colorimetric (diphenylcarbazide),		3500-Cr D	D1687-84(A)		

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
20. Cobalt-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	219.1	3111 B (A or B)	D3558-90	I-3239-84	P.37[11]
AA furnace, or	219.2	3113 B			
Inductively coupled plasma, or	200.7[7]	3120 B			
DCP			D4190-88		Note 34
21. Color, Platinum Cobalt units or dominant wavelength hue, luminance, purity:					
Colorimetric, ADMI	110.1	2120 E			Note 20
Platinum cobalt; or	110.2	2120 B		I-1250-85	
Spectrophotometric	110.3	2120 C			
22. Copper-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	220.1	3111 B or C	D1688-90 (A or B)	I-3271-85 or I-3270-85	974.27[5] p.39[11]
AA furnace,	220.2	3113 B			
Inductively coupled plasma	200.7[7]	3120 B			
DCP,			D4190-88		Note 34
Colorimetric (Neocuproine), or		3500-Cu D or E	D1688-84(88)(A)		
Bicinchoninate					Note 21
23. Cyanide - Total, ug/L:					
Manual distillation with $MgCl_2$		4500-CN-C			
Followed by: titrimetric,		4500-CN-D			p. 22[11]
Manual or	335.2	4500-CN-E	D2036-89(A)	I-3300-85	
Automated[22] spectrophotometric	335.3		D2036-89(A)		
24. Cyanide amenable to chlorination, ug/L: Manual distillation with $MgCl_2$ followed by titrimetric, manual or automated spectrophotometric	335.1	4500-CN-G	D2036-89(B)		
25. Fluoride - Total, mg/L:					
Manual distillation[8]		4500-F-B			
Followed by manual or	340.2	4500-F-C	D1179-88(B)		
automated electrode,				I-4327-85	
SPADNS,	340.1	4500-F-D	D1179-80(A) (1988)		
Or automated complexone	340.3	4500-F-E			
26. Gold Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration	231.1	3111 B			
AA furnace, or	231.2	3113 B			
DCP					Note 34
27. Hardness - Total as $CaCO_3$, mg/L:					
Automated colorimetric,	130.1				
EDTA titration,	130.2	2340 C	D1126-86 (1990)	I-1338-85	973.52B[5]
(or the sum of Ca and Mg as their respective carbonates by ICP or AA direct aspiration)					
(See Parameters 13 and 33)					
28. Hydrogen ion (pH), pH units:					
Electrometric Measurements or	150.1	4500-H^+ B	D1293-84 (A or B) (1990)	I-1586-85	973.41[5]
Automated Electrode					Note 23

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
29. Iridium - Total[6], ug/L:					
Digestion[6] followed by:					
AA direct aspiration	235.1	3111 B			
Or AA furnace	235.2				
30. Iron-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	236.1	3111 B or C	D1068-84 (C or D)	I-3381-84	973.27[5]
AA furnace,	236.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
DCP, or					Note 34
Colorimetric (Phenanthroline)		3500-Fe D	D1068-84(A)		Note 24
31. Kjeldahl nitrogen - Total (as N), mg/L:					
Digestion and distillation	351.3	4500-N org B or C	3590-84(A)		
Followed by titration	351.3	4500-NH_3E	D3590-89)A)		973.46[5]
Nesslerization or	351.3	4500-NH_3C	D3590-89(A)		
Electrode,	351.3	4500-NH_3F or G			
Automated phenate,	351.1	4500-NH_3H		I-4551-78[8]	
Semi-automated block digester,	351.2		D3590-89(B)		
Or potentiometric	351.4		D3590-89(A)		
32. Lead-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	239.1	3111 B or C	D3559-85 (A or B)	I-3399-90	974.27[5]
AA furnace,	239.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
DCP,			D4190-88		Note 34
Voltametry[13] or			D3559-90(C)		
Colorimetric (Dithizone)		3500-Pb D			
33. Magnesium-Total[6], mg/L:					
Digestion[6] followed by:					
Atomic absorption,	242.1	3111 B	D511-88(B)	I-3447-85	974.27[5]
Inductively coupled plasma,	200.7[7]	3120 B			
DCP, or					Note 34
Gravimetric		3500-Mg D	D511-77(A)		
34. Manganese-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	243.1	3111 B or C	D858-90 (A or B)	I-3454-85	974.27[5]
AA furnace,	243.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
DCP,			D4190-88		Note 34
Colorimetric (Persulfate), or		3500-Mn D	D858-84(A)(1988)		920.203[5]
Periodate					Note 25
35. Mercury - Total[6], ug/L:					
Cold vapor, manual or	245.1	3112 B	D3223-86	I-3462-85	977.22[5]
automated	245.2				
36. Molybdenum-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	246.1	3111 D		I-3490-85	
AA furnace,	246.2	3113 B			
Inductively coupled plasma, or	200.7[7]	3120 B			
DCP					Note 34

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
37. Nickel-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	249.1	3111 B or C	D1886-90 (A or B)	I-3499-85	
AA furnace,	249.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
DCP, or			D4190-88		Note 34
Colorimetric (Heptoxime)		3500-Ni D			
38. Nitrate (as N), mg/L:					
Brucine sulfate, or	352.1		D992-71		973.50[5] 419D [19]
Nitrate-nitrite N minus Nitrite N (see parameters 39 and 40)					P. 28[11]
39. Nitrate-nitrite (as N), mg/L:					
Cadmium reduction, manual	353.3	4500-NO_3 E	D867-90(B)		
Or automated, or	353.2	4500-NO_3 F	D3867-90(A)	I-4545-85	
automated hydrazine	353.1	4500-NO_3 H			
40. Nitrite (as N), mg/L:					
Spectrophotometric, manual or	354.1	4400-NO_2 B	D1254-67		Note 27
automated (Diazotization)				I-4540-85	
41. Oil and grease-Total recoverable, mg/L:					
Gravimetric (extraction)	413.1	5520 B			
42. Organic carbon - Total (TOC), mg/L:					
Combustion or oxidation	415.1	5310 B	D2579-85 (A or B)		973.47[5] p. 14[26]
43. Organic nitrogen (as N), mg/L:					
Total Kjeldahl N (Parameter 31)					
minus ammonia N (Parameter 4)					
44. Orthophosphate (as P), mg/L:					
Ascorbic acid method,	365.1	4500-P F		I-4601-85	973.56[5]
automated					
Or manual single reagent or	365.2	4500-P E	D515-88(A)		973.55[5]
Manual two reagent	365.3				
45. Osmium - Total[6], ug/L:					
Digestion[6] followed by:					
AA direct aspiration, or	252.1	3111 D			
AA furnace	252.2				
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification)	360.2	4500-O C	D888-81(C) (1988)	I-1575-78[10]	973.45B[5]
Or electrode	360.1	4500-O G		I-1576-78[10]	
47. Palladium-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	253.1	3111 B			P.S27[11]
AA furnace, or	253.2				P.S28[11]
DCP					Note 34
48. Phenols, ug/L:					
Manual distillation[28]	420.1		D1783-80 (A or B)		Note 29
Followed by manual	420.1				
Or automated[22] colorimetric (4AAP)	420.2				Note 29
49. Phosphorus (elemental), mg/L:					
Gas-Liquid chromatography					Note 30

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
50. Phosphorus - Total, mg/L:					
Persulfate digestion	365.2	4500-P B,5			973.55[5]
Followed by manual or	365.2 or 365.3	4500-P E	D515-88 (A)		
Automated ascorbic acid Reduction, or semi-automated block digestor	365.1 365.4	4500-P F		I-4600-85	973.56[5]
51. Platinum-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	255.1	3111 B			
AA furnace, or	255.2				
DCP					Note 34
52. Potassium - Total[6], mg/L:					
Digestion[6] followed by:					
Atomic absorption,	258.1	3111 B		I-3620-85	973.53[5]
Inductively coupled plasma,	200.7[7]	3120 B			
Flame photometric, or		3500-K D	D1428-82(A)		
Colorimetric (cobaltinitrate)					317B[19]
53. Residue - total, mg/L:					
Gravimetric 103-105°C	160.3	2540 B		I-3750-85	
54. Residue - filterable, mg/L:					
Gravimetric, 180°C	160.1	2540 C		I-1750-85	
55. Residue - nonfilterable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue	160.2	2540 D		I-3765-85	
56. Residue - settleable, mg/L:					
Volumetric (Imhoff cone) or gravimetric	160.5	2540 F			
57. Residue - volatile mg/L:					
Gravimetric, 550°C	160.4	2540 E		I-3753-85	
58. Rhodium - Total[6], ug/L:					
Digestion[6] followed by:					
AA direct aspiration	265.1	3111 B			
Or AA furnace	265.2				
59. Ruthenium - Total[6] ug/L:					
Digestion[6] followed by:					
AA direct aspiration	267.1	3111 B			
Or AA furnace	267.2				
60. Selenium - Total[6] ug/L:					
Digestion[6] followed by:					
AA furnace,	270.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
or AA (gaseous hydride)		3114 B	D3859-88(A)	I-3667-85	
61. Silica - Dissolved, mg/L:					
0.45 micron filtration:					
Followed by manual or automated colorimetric (Molybdosilicate), or	370.1	4500-Si D	D859-88(B)	I-1700-85 I-2700-85	
Inductively coupled plasma	200.7				
62. Silver-Total[31], mg/L:					

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
Digestion[6] followed by:					
AA direct aspiration,	272.1	3111 B or C		I-3720-85	973.27[5] p 37[11]
AA furnace,	272.2	3113 B			
Colorimetric (Dithizone),					319B[19]
Inductively coupled plasma, or	200.7[7]	3120 B			
DCP					Note 34
63. Sodium-Total[6], mg/L:					
Digestion[6] followed by:					
Atomic absorption,	273.1	3111 B		I-3735-85	973.54[4]
Inductively coupled plasma,	200.7[7]	3120 B			
DCP, or					Note 34
Flame photometric		3500-Na D	D1428-82(A)		
64. Specific conductance, micromhos/cm at 25°C: Wheatstone bridge	120.1	2510 B	D1125-82(A)	I-1780-85	973.40[5]
65. Sulfate (as SO4), mg/L:					
Automated colorimetric (barium chloroanilate),	375.1				
Gravimetric, or	375.3	4500-SO_4^{2-} C or D	D516-82(A) (1988)		925.54[5]
Turbidimetric	375.4		D516-88		426C[32]
66. Sulfide (as S), mg/L:					
Titrimetric (iodine) or	376.1	4500-S^{2-} E		I-3840-85	228A[33]
Colorimetric (methylene blue)	376.2	4500-S^{2-} D			
67. Sulfite (as SO_3), mg/L:					
Titrimetric (iodine-iodate)	377.1	4500-SO_3^{2-}	D1339-84(C)		
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1	5540 C	D2330-88		
69. Temperature, °C: Thermometric	170.1	2550 B			Note 34
70. Thallium - Total[6], ug/L:					
Digestion[6] followed by:					
AA direct aspiration,	279.1	3111 B			
AA furnace, or	279.2	3113 B			
Inductively coupled plasma	200.7[7]				
71. Tin - Total[6], ug/L:					
Digestion[6] followed by:					
AA direct aspiration or	282.1	3111 B		I-3850-78[10]	
AA furnace	282.2	3113 B			
72. Titanium-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration ,	283.1	3111 D			
AA furnace, or	283.2	3113 B			
DCP					Note 34
73. Turbidity, NTU: Nephelometric	180.1	2130 B	D1889-88a	I-3860-85	
74. Vanadium-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	286.1	3111 D			
AA furnace,	286.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
DCP, or			D4190-88		Note 34
Colorimetric (Gallic acid)		3500-V D	D3373-84(A) (1988)		

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA[1]	Standard Methods[4]	ASTM[B]	USGS[1*]	Other
75. Zinc-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,	289.1	3111 B or C	D1691-90(A or B)	I-3900-85	974.27[5] P.37[11]
AA furnace,	289.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
DCP,			D4190-88		Note 34
Colorimetric (Dithizone), or		3500-Zn E			
Colorimetric (Zincon)		3500-Zn F			Note 34

TABLE B NOTES

[1] "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 United States Environmental Protection Agency,Revised March 1983 and 1979 where applicable. Available from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

[2] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 17th Edition, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[3] "1991 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1986. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[4] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments", U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1989, unless otherwise stated. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] "Official Methods of Analysis of the Association of Official Analytical Chemists", methods manual, 15th Edition (1990). Available from The Association of Official Analytical Chemists, 1111 N. 19th Street, Suite 210, Arlington, VA 22209.

[6] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979". One (4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrocholoric acids (4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instructions and/or cautions.

Note: If the digestion included in one of the other approved references is different than the above, the EPA procedure will be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals will be followed. Sample digestion for dissolved metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample solution to be analyzed meets the following criteria:

(a) has a low COD (<20),
(b) is visibly transparent with a turbidity measurement of 1 NTU or less,
(c) is colorless with no perceptible odor, and
(d) is of one liquid phase and free of particulate or suspended matter following acidification.

[7] The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes", is given in Appendix C of the Federal Register, October 26, 1984 (Part VIII, 40 CFR part 136). Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 212002.

[8] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[9] Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnalyzerII. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.

[10] The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979). Available on inter-library loan.

[11] "American National Standard on Photographic Processing Effluents", April 2, 1975. Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.

[12] "Selected Analytical Methods Approved and cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of "Standard Methods for the Examination of Water and Wastewater," from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1981. Available on inter-library loan.

[13] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[14] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required, can the permittee report data obtained using the nitrification inhibitor.

[15] OIC Chemical Oxygen Demand Method. Available from Oceanography International Corporation, 512 West loop, P.O. Box 2980, College Station, TX 77840.

[16] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[17] The back titration method will be used.

[18] ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Available from Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.

[19] The approved method is that cited in the "Standard Methods for the Examination of Water and Wastewater", 14th Edition, 1976. Available on inter-library loan.

[20] "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color", NCASI Technical Bulletin No. 253. December, 1971. Available from National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016.

[21] Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[22] After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.03 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[23] Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.

[24] 1, 10-Phenanthroline Method for Iron, Hach Method 8008, 1980. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[25] Periodate Oxidation Method for Manganese, Method 8034. Hach Handbook of Wastewater Analysis, 1979, pp. 2-113 and 2-117. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[26] "Methods for Analysis of Organic Substances in Water", by D. F. Goerlitz and Eugene Brown: USGS-TWRI, Book 5, Chapter A3, p. 4, 1972. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[27] Nitrite Nitrogen, Hach Method 8507. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[28] Just prior to distillation, adjust the sulfuric acid preserved sample to pH 4 with 1 + 9 NaOH.

[29] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure. Available on inter-library loan.

[30] "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", by R. F. Addison and R. G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

[31] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L,

20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[32] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 15th Edition. Available on inter-library loan.

[33] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 13th Edition. Available on inter-library loan.

[34] "Water Temperature-Influential Factors, Field Measurement, and Data Presentation", by H. H. Stevens, Jr., J. Ficke, and G. F. Smoot: USGS-TWRI Book 1, Chapter D1, 1975. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[35] Zincon Method of Zinc Method 8009. Hach Handbook for Water Analysis, 1979, pp. 2-231 and 2-333. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[36] "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986-Revised 1991, Applied Research Laboratories, Inc., 24911 Avenue Stanford, Valencia, CA 91355.

TABLE C
List of Approved Test Procedures for Non-Pesticide Organic Compounds

EPA METHOD NUMBER [2,7]

	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
1.	Acenaphthene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
2.	Acenaphthylene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
3.	Acrolein	603	[4]624, 1624				
4.	Acrylonitrile	603	[4]624, 1624	610			
5.	Anthracene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
6.	Benzene	602	624, 1624		6210 B, 6220 B		
7.	Benzidine		[5]625, 1625	605			
8.	Benzo(a)anthracene	610	625, 1625	610	6410 B; 6440 B	D4657-87	
9.	Benzo(a)pyrene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
10.	Benzo(b)fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
11.	Benzo(g,h,i)perylene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
12.	Benzo(k)fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
13.	Benzyl chloride						Note 3, p. 130; Note 6, p. S102
14.	Benzyl butyl phthalate	606	625, 1625		6410 B		
15.	Bis(2-chloroethoxy) methane	611	625, 1625		6410 B		
16.	Bis(2-chloroethyl)ether	611	625, 1625		6410 B		
17.	Bis(2-ethylhexyl)phthalate	606	625, 1625		6410 B, 6230 B		
18.	Bromodichloromethane	601	624, 1624		6410 B, 6230 B		
19.	Bromoform	601	624, 1624		6410 B, 6230 B		
20.	Bromomethane	601	624, 1624		6410 B, 6230 B		
21.	4-Bromophenylphenyl ether	611	625, 1625		6410 B		
22.	Carbon tetrachloride	601	624, 1624		6230 B, 6410 B		Note 3, p. 130
23.	4-Chloro-3-methylphenol	604	625, 1625		6410 B, 6420 B		
24.	Chlorobenzene	601 602	624, 1624		6210 B, 6220 B, 6230 B		Note 3, p. 130
25.	Chloroethane	601	624, 1624		6210 B, 6230 B		
26.	2-Chloroethylvinyl ether	601	624, 1624		6210 B, 6230 B		
27.	Chloroform	601	624, 1624		6210 B, 6230 B		Note 3, p. 130
28.	Chloromethane	601	624, 1624		6210 B, 6230 B		
29.	2-Chloronaphthalene	612	625, 1625		6410 B		
30.	2-Chlorophenol	604	625, 1625		6410 B, 6420 B		
31.	4-Chlorophenylphenyl ether	611	625, 1625		6410 B		
32.	Chrysene	610	625, 1625	610	6410 B, 6440 B	D4656-87	

TABLE C (continued)
List of Approved Test Procedures for Non-Pesticide Organic Compounds

		EPA METHOD NUMBER [2,7]					
	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
33.	Dibenzo(a,h)anthracene	610	625 1625	610	6410 B, 6440 B	D4656-87	
34.	Dibromochloromethane	601	624 1624	610	6410 B, 6440 B	D4656-87	
35.	1,2-Dichlorobenzene	601 602 612	624 625 1625		6410 B, 6440 B, 6220 B		
36.	1,3-Dichlorobenzene	601 602 612	624 625 1625		6410 B, 6440 B, 6220 B		
37.	1,4-Dichlorobenzene	601 602 612	624 625 1625		6410 B, 6440 B, 6220 B		
38.	3,3-Dichlorobenzidine		625 1625	605	6410 B		
39.	Dichlorodifluoromethane	601			6230 B		
40.	1,1-Dichloroethane	601	624 1624		6230 B, 6210 B		
41.	1,2-Dichloroethane	601	624 1624		6230 B, 6210 B		
42.	1,1-Dichloroethene	601	624 1624		6230 B, 6210 B		
43.	trans-1,2-Dichloroethene	601	624 1624		6230 B, 6210 B		
44.	2,4-Dichlorophenol	604	625 1625		6420 B, 6410 B		
45.	1,2-Dichloropropane	601	624 1624		6230 B, 6210 B		
46.	cis-1,3 Dichloropropene	601	624 1624		6230 B, 6210 B		
47.	trans-1,3-Dichloropropene	601	624 1624		6230 B, 6210 B		
48.	Diethyl phthalate	606	625 1625		6410 B		
49.	2,4-Dimethlyphenol	604	625 1625		6420 B, 6410 B		
50.	Dimethyl phthalate	606	625 1625		6410 B		
51.	Di-n-butyl phthalate	606	625 1625		6410 B		
52.	Di-n-octyl phthalate	606	625 1625		6410 B		
53.	2,4-Dinitrophenol	604	625 1625		6420 B, 6410 B		
54.	2,4-Dinitrotoluene	609	625 1625		6410 B		
55.	2,6-Dinitrotoluene	609	625 1625		6410 B		
56.	Epichlorohydrin						Note 3, p. 130;Note 6, p. S102
57.	Ethylbenzene	602	624 1624		6220 B, 6210 B		
58.	Fluoranthene	610	625 1625	610	6410 B, 6440 B	D4657-87	
59.	Fluorene	610	625 1625	610	6410 B, 6440 B	D4657-87	
59e.	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin		1613A[8]				
59m.	1,2,3,4,6,7,8-Heptachlorodibenzofuran		1613A[8]				
59t.	1,2,3,4,7,8,9-Heptachlorodibenzofuran		1613A[8]				
60.	Hexachlorobenzene	612	625 1625		6410 B		
61.	Hexachlorobutadiene	612	625 1625		6410 B		
62.	Hexachlorocyclopentadiene	612	5625 1625		6410 B		
62c	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin		1613A[8]				
62f.	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin		1613A[8]				
62i.	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin		1613A[8]				
62m.	1,2,3,4,7,8-Hexachlorodibenzofuran		1613A[8]				
62p.	1,2,3,6,7,8-Hexachlorodibenzofuran		1613A[8]				
62s.	1,2,3,7,8,9-Hexachlorodibenzofuran		1613A[8]				
62v.	2,3,4,6,7,8-Hexachlorodibenzofuran		1613A[8]				
63.	Hexachloroethane	612	625 1625		6410 B		

TABLE C (continued)
List of Approved Test Procedures for Non-Pesticide Organic Compounds

		EPA METHOD NUMBER [2,7]					
	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
64.	Ideno (1,2-3-cd)pyrene	610	625 1625	610	6410 B, 6440 B	D4657-87	
65.	Isophorone	609	625 1625		6410 B		
66.	Methylene chloride	601	624 1624		6230 B		Note 3, p. 130
67.	2-Methyl-4,6-dinitrophenol	604	625 1625		6420 B, 6410 B		
68.	Naphthalene	610	625 1625	610	6410 B, 6440 B		
69.	Nitrobenzene	609	625 1625		6410 B	D4657-87	
70.	2-Nitrophenol	604	625 1625		6410 B, 6420 B		
71.	4-Nitrophenol	604	625 1625		6410 B, 6420 B		
72.	N-Nitrosodimethylamine	607	5625 1625		6410 B		
73.	N-Nitrosodi-n-propylamine	607	625 1625		6410 B		
74.	N-Nitrosodiphenylamine	607	[5]625 1625		6410 B		
74h.	Octachlorodibenzo-p-dioxin		1613A[8]				
74r.	Octachlorodibenzofuran		1613A[8]				
75.	2,2-Oxybis (1-chloropropane)	611	625 1625		6410 B		
76.	PCB-1016	608	625		6410 B		Note 3, p.43
77.	PCB-1221	608	625		6410 B		Note 3, p.43
78.	PCB-1232	608	625		6410 B		Note 3, p.43
79.	PCB-1242	608	625		6410 B		Note 3, p.43
80.	PCB-1248	608	625				
81.	PCB-1254	608	625		6410 B		Note 3, p.43
82.	PCB-1260	608	625		6410 B, 6630 B		Note 3, p.43
82e.	1,2,3,7,8-Pentachlorodibenzo-p-dioxin		1613A[8]				
82m.	1,2,3,7,8-Pentachlorodibenzofuran		1613A[8]				
82t.	2,3,4,7,8-Pentachlorodibenzofuran		1613A[8]				
87m.	2,3,7,8-Tetrachlorodibenzofuran		1613A[8]				
83.	Pentachlorophenol	604	625 1625		6410 B, 6630 B		Note 3, p.140
84.	Phenanthrene	610	625 1625	610	6410 B, 6440 B	D4657-87	
85.	Phenol	604	625 1625		6420 B, 6410 B		
86.	Pyrene	610	625 1625	610	6410 B, 6440 B	D4657-87	
87.	2,3,7,8-Tetrachlorodibenzo-p-dioxin			[5a]613, 1613 A			
88.	1,1,2,2-Tetrachloroethane	601	624 1624		6230 B, 6210 B		Note 3, p.130
89.	Tetrachloroethene	601	624 1624		6230 B, 6210 B		Note 3, p.130
90.	Toluene	602	624 1624		6210 B, 6220 B		
91.	1,2,4-Trichlorobenzene	612	625 1625		6410 B		Note 3, p.130
92.	1,1,1-Trichloroethane	601	624 1624		6210 B, 6220 B		
93.	1,1,2-Trichloroethane	601	624 1624		6210 B, 6220 B		Note 3, p.130
94.	Trichloroethene	601	624 1624		6210 B, 6230 B		
95.	Trichlorofluoromethane	601	624		6210 B, 6230 B		
96.	2,4,6-Trichlorophenol	604	625 1625		6410 B, 6240 B		
97.	Vinyl chloride	601	624 1624		6210 B, 6230 B		

TABLE C NOTES

[1] All parameters are expressed in micrograms per liter (µg/L).

[2] The full text of Methods 601-613, 624, 625, 1624, and 1625, are given in Appendix A of 40 CFR part 136, "Test Procedures for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit." Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethyamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

[5a] 625 Screening only.

[6] "Selected Analytical Methods approved and Cited by the United States Environmental Protection Agency," Supplement to the 15th Edition of "Standard Methods for the Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1613A, 1624, and 1625 in accordance with procedures in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

[8] Method 1613 Revision A: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution, HRGC/HRMS, Environmental Protection Agency, Federal Register, page 5098, February 1991. Available from the Superintendent of Documents, US Government Printing Office, Washington, D.C. 20402.

[9] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 17th Edition, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[10] "1991 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1986. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

TABLE D
List of Approved Test Procedures for Pesticides[1]

Parameter (micrograms per liter)	Method	EPA[2,7]	Standard[4] Methods	ASTM[8]	Other

TABLE D (continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (micrograms per liter)		Method	EPA[2,7]	Standard[4] Methods	ASTM[B]	Other
1.	Aldrin	GC	608	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
		GC/MS	625	6410 B		
2.	Ametryn	GC				Note 3, p. 83; Note 6, p. S68.
3.	Aminocarb	TLC				Note 3, p. 94; Note 6, p. S16.
4.	Atraton	GC				Note 3, p. 83; Note 6, p. S68.
5.	Atrazine	GC				Note 3. p. 83; Note 6, p. S68.
6.	Azinphos methyl	GC				Note 3. p. 25; Note 6, p. S51.
7.	Barban	TLC				Note 3, p. 104; Note 6, p. S64.
8.	α-BHC	GC	608	6630 B & C	D3086-90	Note 3, p. 7.
		GC/MS	[5]625	6410 B		
9.	β-BHC	GC	608	6630 C	D3086-90	
		GC/MS	625	6410 B		
10.	δ-BHC	GC	608	6630 C	D3086-90	
		GC/MS	[5]625	6410 B		
11.	-BHC (Lindane)	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625			
12.	Captain	GC		6630 B	D3086-90	Note 3. p. 7.
13.	Carbaryl	TLC				Note 3. p. 94; Note 6. p. S60.
14.	Carbophenothion	GC				Note 4, p. 30; Note 6, p. S73.
15.	Chlordane	GC	608	6630 B & C	D3086-90	Note 3, p. 7
		GC/MS	625			
16.	Chloropropham	TLC				Note 3, p. 104; Note 6, p. S64.
17.	2,4-D	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
18.	4,4'-DDD	GC	608	6630 B & C	D3086-90	Note 3. p. 7; Note 4, p. 30.
		GC/MS	625	6410 B		
19.	4,4'-DDE	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
		GC/MS	625	6410 B		
20.	4,4'-DDT	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625	6410		
21.	Demeton-O	GC				Note 3, p. 25; Note 6, p. S51.
22.	Demeton-S	GC				Note 3, p. 25; Note 6, p. S51.
23.	Diazinon	GC				Note 3, p. 25; Note 4, p. 30;
					Note 6 p. S51	
24.	Dicamba	GC				Note 3, p. 115.
25.	Dichlofenthion	GC				Note 4, p. 30; Note 6, p. S73.
26.	Dichloran	GC		6630 B & C	D3086-90	Note 3, p. 7.
27.	Dicofol	GC				
28.	Dieldrin	GC	608	6630 B & C		Note 3, p. 7; Note 4, p. 30.
		GC/MS	625	6410 B		
29.	Dioxathion	GC				Note 4, p. 30; Note 6, p. S73.
30.	Disulfoton	GC				Note 3, p. 25; Note 6, p. S51.
31.	Diuron	TLC				Note 3, p. 104; Note 6, p. S64.

TABLE D (continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (micrograms per liter)		Method	EPA[2,7]	Standard[4] Methods	ASTM[B]	Other
32.	Endosulfan I	GC	608	6630 B & C	D3086-90	Note 3, p. 7.
		GC/MS	[5]625	6410 B		
33.	Endosulfan II	GC	608	6630 B & C	D3086-90	Note 3, p. 7.
		GC/MS	[5]625	6410 B		
34.	Endosulfan sulfate	GC	608	6630 C		
		GC/MS	625	6410 B		
35.	Endrin	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
		GC/MS	[5]625	6410 B		
36.	Endrin aldehyde	GC	608	6630 B & C	D3086-90	
		GC/MS	625	6410		
37.	Ethion	GC				Note 4, p. 30; Note 6, p. S73.
38.	Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39.	Fenuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40.	Heptachlor	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625	6410 B		
41.	Heptachlor epoxide	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30; Note 6 p.S73
		GC/MS	625	6410 B		
42.	Isodrin	GC				Note 4, p. 30; Note 6, p. S73.
43.	Linuron	TLC				Note 3, p. 104; Note 6, p. S64
44.	Malathion	GC		6630 C		Note 3, p. 25; Note 4, p.30; Note 6, p.S51
45.	Methiocarb	TLC				Note 3, p. 94; Note 6, p. S60.
46.	Methoxychlor	GC		6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
47.	Mexacarbate	TLC				Note 3, p. 94; Note 6, p. S60.
48.	Mirex	GC		6630 B & C		Note 3, p. 7.
49.	Monuron	TLC				Note 3, p. 104; Note 6, p. S64
50.	Monuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
51.	Neburon	TLC				Note 3, p. 104; Note 6, p. S64.
52.	Parathion methyl	GC		6630 C		Note 3, p. 25; Note 4, p. 30.
53.	Parathion ethyl	GC		6630 B & C	D3086-90	Note 3, p. 25.
54.	PCNB	GC		6630 B & C		Note 3, p. 7.
55.	Perthane	GC			D3086-90	
56.	Prometron	GC				Note 3, p. 83; Note 6, p. S68.
57.	Prometron	GC				Note 3, p. 83; Note 6, p. S68.
58.	Propazine	GC				Note 3, p. 83; Note 6, p. S68.
59.	Propham	TLC				Note 3, p. 104; Note 6, p. S64
60.	Propoxur	TLC				Note 3, p. 94; Note 6, p. S60.
61.	Secbumeton	TLC				Note 3, p. 83; Note 6, p. S68.
62.	Siduron	TLC				Note 3, p. 104; Note 6, p. S64
63.	Simazine	GC				Note 3, p. 83; Note 6, p. S68.
64.	Strobane	GC		6630 B & C		Note 3, p. 7.
65.	Swep	TLC				Note 3, p. 104; Note 6, p. S64.
66.	2,4,5-T	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
67.	2,4,5-TP (Silvex)	GC		6640 B		Note 3, p. 115.

TABLE D (continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (micrograms per liter)		Method	EPA[2,7]	Standard[A] Methods	ASTM[B]	Other
68.	Terbuthylazine	GC				Note 3, p. 83; Note 6, p. S68.
69.	Toxaphene	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625	6410 B		
70.	Trifluralin	GC		6630 B		Note 3, p. 7.

TABLE D NOTES

[A] "Standard Methods for the Examination of Water and Wastewater", 17th Edition, Joint Editorial Board, American Public Heath Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[B] "1991 Annual Book of Standards, "Water" Section 11, American Society for Testing and Materials, 1980. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table C, where entries are listed by chemical name.

[2] The full text of methods 608 and 625 are given in Appendix A of the Federal Register, October 26, 1984 (Part VIII, 40 CFR part 136), "Test Procedure for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit". Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater". U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[4] "Methods for Analysis of Organic Substances in Water", Book 5, Chapter A3, 1987. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] The method may be extended to include a(alpha)-BHC, d(delta)-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of "Standard Methods for Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 15th St., N.W., Washington, D.C. 20005.

[7] Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A in 40 CFR part 136 in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

TABLE E
List of Approved Radiological Test Procedures

Parameter and Units	Method	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]

3.	Beta-Total, pCi per liter	Proportional counter	900.0	703	D1943-81 pp. 75 and 78[5]
4.	Beta-Counting error, pCi	Proportional counter	Appendix B	703	D1943-81 p. 79
5.	(a) Radium-Total	Proportional counter	903.0	705	D2460-70
	(b) ^{226}Ra, pCi per liter	Scintillation counter	903.1	706	D3454-79 p. 81

TABLE E NOTES

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/-4-80-032 (1980, U.S. Environmental Protection Agency, August 1980.

[2] "Standard Methods for the Examination of Water and Wastewater", 17th Edition, Joint Editorial Board, American Public Heath Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[3] "1991 Annual Book of Standards, "Water" Section 11, American Society for Testing and Materials, 1980. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[4] "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," . U.S. Geological Survvey, Open-File Report 76-177 (1976)

[5] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total".

TABLE F

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name		Container[1]	Preservation[2,3]	Maximum holding time[4]
TABLE A - Bacterial Tests:				
1-5.	Bacteria	P,G	Cool, 4°C, 0.008%, Na_2, S_2, O_3[5]	6 hours.
6-7.	Enteroviruses	P,G	Cool, 4°C	24 hours
8.	Mutagenicity	G, Teflon-lined cap	Cool, 4°C	7 days
9-12.	Acute & chronic toxicity	P,G	Cool, 4°C	48 hours
TABLE B - Inorganic Tests:				
1.	Acidity	P,G	Cool, 4°C	14 days
2.	Alkalinity	P,G	Cool, 4°C	14 days
4.	Ammonia	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
9.	Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
11.	Bromide	P,G	None required	28 days
14.	Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
15.	Chemical oxygen demand	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
16.	Chloride	P,G	None required	28 days
17.	Chlorine, total residual	P,G	None required	Analyze immediately
21.	Color	P,G	Cool, 4°C	48 hours
23-24.	Cyanide, total and amenable to chlorination	P,G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid[5]	14 days[6]
25.	Fluoride	P	None required	28 days
27.	Hardness	P,G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
28.	Hydrogen ion (pH)	P,G	None required	Analyze immediately
31,43.	Kjeldahl and organic nitrogen	P,G	Cool, 4°C H_2SO_4 to pH<2	28 days
Metals[7]:				
18.	Chromium VI	P,G	Cool, 4°C	24 hours
35.	Mercury	P,G	HNO_3 to pH<2	28 days
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals except chromium VI and mercury		P,G	HNO_3 to pH<2	6 months
38.	Nitrate	P,G	Cool, 4°C	48 hours
39.	Nitrate-nitrite	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
40.	Nitrite	P,G	Cool, 4°C	48 hours
41.	Oil and grease	G	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
42.	Organic carbon	P,G	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
44.	Orthophosphate	P,G	Filter immediately, Cool, 4°C	48 hours
46.	Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately
47.	Winkler	G Bottle and top	Fix on site and store in dark	8 hours
48.	Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2	28 days
49.	Phosphorus (elemental)	G	Cool, 4°C	48 hours
50.	Phosphorus, total	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days

Parameter No./name		Container[1]	Preservation[2,3]	Maximum holding time[4]
53.	Residue, total	P,G	Cool, 4°C	7 days
54.	Residue, Filterable	P,G	Cool, 4°C	7 days
55.	Residue, Nonfilterable (TSS)	P,G	Cool, 4°C	7 days
56.	Residue, Settleable	P,G	Cool, 4°C	48 hours
57.	Residue, Volatile	P,G	Cool, 4°C	7 days
61.	Silica	P	Cool, 4°C	28 days
64.	Specific conductance	P,G	Cool, 4°C	28 days
65.	Sulfate	P,G	Cool, 4°C	28 days
66.	Sulfide	P,G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9	7 days
67.	Sulfite	P,G	None required	Analyze immediately
68.	Surfactants	P,G	Cool, 4°C	48 hours
69.	Temperature	P,G	None required	Analyze immediately
73.	Turbidity	P,G	Cool, 4°C	48 hours
TABLE C - Organic Tests[8]				
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97. Purgeable Halocarbons		G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	14 days
6, 57, 90. Purgeable aromatic		G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] HCl to pH<2[9]	14 days
3, 4. Acrolein and acrylonitrile		G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], Adjust pH to 4-5[10]	14 days
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols[11]		G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction. 40 days after extraction.
7, 38. Benzidines[11]		G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
14 17, 48, 50-52. Phthalate esters[11]		G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
72-74. Nitrosamines[11,14]		G, Teflon-lined cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
76-82. PCBs[11] acrylonitrile		G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
54, 55, 65, 69. Nitroaromatics and isophorone[11]		G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark	7 days until extraction; 40 days after extraction.
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons[11]		G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark	7 days until extraction; 40 days after extraction.
15, 16, 21, 31, 75. Haloethers[11]		G. Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
29, 35-37, 60-63, 91. Chlorinated hydrocarbons[11]		G. Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
59e-59t, 62c-62v, 74h-74r, 82e-82t, 87, 87m. Chlorinated Dioxins and Furans.		G. Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
TABLE D - Pesticide Tests:				
1-70. Pesticides[11]		G. Teflon-lined cap	Cool, 4°C, pH 5-9[15]	7 days until extraction; 40 days after extraction.
TABLE E - Radiological Tests				
1-5 Alpha, beta, and radium		P,G	HNO_3 to pH<2	6 months

TABLE F NOTES:

[1] Polyethylene (P) or Glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic)

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed.

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

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Virus samples can be stored indefinitely at -70°C. Samples used for toxicity tests are to be used for test initiation or for renewal of test solutions within 36 hours of collection as grab samples or after removal from composite samplers. Samples for biological or chemical analysis may be held for longer periods than specified in this table only if the permittee or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator. s. NR 219.06 some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

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

[9] Samples receiving no pH adjustment must be analyzed within seven days of sampling.

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

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

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

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O^3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

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

SECTION 80. NR 347.06 (5) and (6) are amended to read:

(5) Sample handling after collection and prior to analysis. Sample handling and storage prior to analysis shall be in accordance with ~~methods from applicable sources enumerated in ch. NR 149~~ the maximum holding times and container types given in table F of ch. NR 219. Samples shall be preserved at the time of collection by cooling to 4°C.

(6) Analyses to be performed on sediment samples. Analyses shall be done in accordance with methods from applicable sources enumerated in ch. NR 149. Analyses submitted to the department under this chapter shall be done by a laboratory certified or registered under ch. NR 149.

The foregoing rules were approved and adopted by the State of Wisconsin Natural Resources Board on June 25, 1992.

Sections 1-15, 17, 18, 21, 22, 24, 27, 29, 32, 35-43, 46, 49, 51, 52, 56, 58, 60, 60-62, 64, 65, 69-77, 79, and 80 of the rule contained herein shall take effect on the first day of the month following publication in the Wisconsin administrative register as provided in s. 227.22 (2)(intro.), Stats. Sections 16, 20, 23, 26, 28, 30, 31, 34, 44, 45, 47, 48, 50, 53-55, 57, 59, 66, and 68 of the rule shall take effect on January 1, 1993. Sections 19, 33, 63, 67, and 78 of the rule shall take effect on July 1, 1993.

Dated at Madison, Wisconsin September 21, 1992

STATE OF WISCONSIN DEPARTMENT OF NATURAL RESOURCES

By Carroll D. Besadny

Carroll D. Besadny, Secretary