CR 93-149

WISCONSIN
DEPT. OF NATURAL RESOURCES
George E. Meyer
Secretary

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

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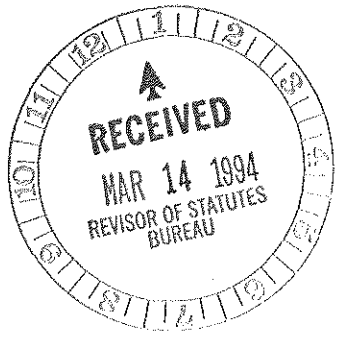
STATE OF WISCONSIN)
) ss
DEPARTMENT OF NATURAL RESOURCES)



TO ALL TO WHOM THESE PRESENTS SHALL COME, GREETINGS:

I, George E. Meyer, 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-26-93 was duly approved and adopted by this Department on December 9, 1993. 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.

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 3rd day of March, 1994.

George E. Meyer
George E. Meyer, Secretary

(SEAL)

5-1-94

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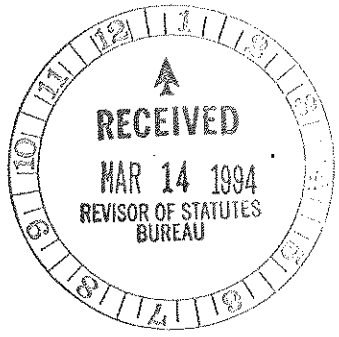
ORDER OF THE STATE OF WISCONSIN NATURAL RESOURCES BOARD TO REPEAL, RENUMBER & AMEND, AMEND, REPEAL & RECREATE AND CREATE RULES

IN THE MATTER of repealing ss. NR 149.21(7) and 149.46; renumbering ss. NR 149.21(1) to (6) and (8) and amending as renumbered ss. NR 149.21(1), (6) and (7); amending ss. NR 149.03(5)(c), 149.04, Table 1, items 7, 15, and 18, 149.13(11), 149.21 (title), 149.42(1)(d)2, 219.04(1), 219.04, Table A, (title), items 1 & 9, 219.04, table B, (title), items 3, 5, 6, 7, 8, 9, 10, 12, 13, 18, 19, 20, 22, 26, 30, 32, 33, 34, 36, 37, 47, 51, 60, 62, 63, 72, 74, and 75, 219.04, Table B Note [7] and [36], 219.04, Table C, (title), 219.04, Table D, (title), 219.04, Table E, (title), 219.04, Table F, (title), and 605.08(5)(a); repealing & recreating ss. NR 149.05(1), 149.42(2), 149.43(title), (1) and (2), and 219.04, Table B, Note [6]; and creating ss. NR 149.03(4m), 149.03(16m), 149.07(1g), 149.07(5), 149.21(8), 149.42(1)(a)8 and (b)9, 149.43(3)(title), 219.04 Table B, Notes [6m] and [19m], and 219.04, Table EM; of the Wisconsin Administrative Code pertaining to laboratory certification and registration, preservation procedures and laboratory procedures.



TS-26-93

Analysis Prepared by Department of Natural Resources

Statutory authority: ss. 144.62 (8)(c), 144.95 (7), 144.95 (9), and 227.11, Stats.

Statutes interpreted: ss. 144.62, 144.95, and 147.08, Stats.

The effect of amending ch. NR 149, Wis. Adm. Code, is to 1) amend the citation to SW-846 to cite the current addition [SECTION 1], 2) add a definition for analytical staff and MCL [Section 2, 3], 3) add Safe Drinking Water Act (SDWA) Phase V parameters to SDWA Certification [SECTION 4], 4) add certification and registration for total releasable cyanide, total releasable sulfide, corrosivity, synthetic precipitation leaching procedure and delete total recoverable petroleum hydrocarbons [SECTION 4], 5) increase fees for certified and registered laboratories [SECTION 5], 6) add a provision that an application expires after 1 year from receipt, if not completed [SECTION 6], 7) add a provision requiring information on transfer of laboratory ownership [SECTION 7], 8) add requirements pertaining to submittal of initial precision and accuracy for dioxin certification or registration [SECTION 8], 9) correct miscellaneous editorial errors [SECTION 9], 10) clarify and simplify the rule

[SECTIONs 10, 11, & 12], 11) add referral and suspension for laboratories doing testing for those tests that they do not have certification [SECTIONs 13 and 15], 12) add revocation for failure to implement or comply with a quality control program [SECTION 13], 13) clarify hearing procedures [SECTION 14], 14) amend code for approval of 3rd party certification [SECTION 16], 15) add a title for the Health and Social Services agreement [SECTION 17], and 16) delete section NR 149.46 (Procedures for Revising Certification or Registration as a Result of the 1992 Amendments) [SECTION 18].

The effect of amending ch. NR 219, Wis. Adm. Code, is to 1) incorporate changes to the federal rules on page 41830 of the September 11, 1992 Federal Register [SECTIONs 21, 22, & 23] and page 9387 of the February 19, 1993 Federal Register [SECTIONs 19 & 28], 2) delete method 405.1 for BOD_5 [SECTIONs 21], 3) add method 218.6 for dissolved hexavalent chromium by ion chromatography [SECTIONs 21 & 23], 4) correct titles to tables A, B, C, D, E, and F [SECTION 20, 21, 25, 26, 27, 29], and 5) correct miscellaneous editorial errors [SECTIONs 20, 21, & 24].

The effect of amending ch. NR 605.08 (5), Wis. Adm. Code, is to make state rules consistent with federal rules that were amended on page 55114 of the November 24, 1992 Federal Register [Section 30].

SECTION 1. NR 149.03 (5)(c) is amended to read:

NR 149.03 (5)(c) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, November, 1986, including December 1987 and ~~November 1990~~ July 1992 updates.

SECTION 2. NR 149.03(4m) is created to read:

NR 149.03(4m) "Analytical staff" means, but is not limited to, the laboratory director, supervisory staff, quality assurance staff, laboratory technicians and chemists.

SECTION 3. NR 149.03(16m) is created to read:

NR 149.03(16m) "MCL" means the maximum permissible level of a contaminant in water which is delivered to any user of a public water system.

SECTION 4. NR 149.04, Table 1, items 7, 15, and 18 are amended to read:

7.	General III	No reference sample	~~EP~~ Extraction Procedure (EP) Toxicity, Ignitability, Reactivity, Total Releasable Cyanide, Total Releasable Sulfide, Corrosivity, Waste Fingerprinting Analyses, Total Organic Carbon, Total Organic Halide, Toxicity Characteristic Leaching Procedure (TCLP), Synthetic Precipitation Leaching Procedure (SPLP). Note: Certification or registration for EP, TCLP, or SPLP under this test category is only for the extraction and does not include the analytes.
15.	Organics; Petroleum Hydrocarbons	Gasoline Range Organics (GRO), Diesel Range Organics (DRO), ~~Total Recoverable Petroleum Hydrocarbons (TRPH),~~ Petroleum Volatile Organic ~~Carbon~~ Compounds (PVOC).	Gasoline Range Organics, Diesel Range Organics, Petroleum Volatile Organic Compounds (PVOC), ~~and Total Recoverable Petroleum Hydrocarbons (TRPH)~~.
18.	Safe Drinking Water	Each analyte or analyte group for which certification is desired.	~~Arsenic, Asbestos, Barium, Cadmium, Chromium, Copper,~~ Cyanide, Fluoride, ~~Lead, Mercury~~ Metals, Nitrate as Nitrogen, Nitrite as Nitrogen, Nitrate+Nitrite as Nitrogen, ~~Selenium, Alachlor, Atrazine, Carbofuran, Chlordane, Dibromochloropropane, Endrin, Ethylene Dibromide , Heptachlor, Heptachlor Epoxide, Lindane, Methoxychlor, Polychlorinated Biphenyls, Toxaphene, 2,4-D, 2,4,5, TP,~~ Synthetic Organic Contaminants, Total Trihalomethanes, ~~Benzene, Vinyl Chloride, Carbon Tetrachloride, 1,2 Dichloroethane,~~

~~Trichloroethylene, 1,1 Dichloroethylene, 1,1,1 Trichloroethane, para-Dichlorobenze, 1,2-Dichloropropane, Ethylbenzene, Chlorobenzene, o-Dichlorobenzene, Styrene, Tetrachloroethylene, Toluene, Trans 1,2 Dichloroethylene, Cis-1,2 Dichloroethylene, Xylenes~~ Volatile Organics.

~~Note: Federal regulations include cis-1,2-dichloroethylene.~~
Note: The list of certifiable parameters within each group is given on the application form.

SECTION 5. NR 149.05 (1) is repealed and recreated to read:

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

(a) The total fee income shall be designed to generate revenues equal to the approved spending authority for this program. The department may increase the fees given in Table 2 by no more than the previous year's consumer price index.

Note: The approved spending authority is given in s. 20.370 (2) (fj), Stats., and may be revised by the legislative joint committee on finance to cover additional program cost.

(b) The minimum annual certification fee for laboratories certified in any of the test categories 5 through 19 is $600. There is no minimum fee for registration. The department may adjust this fee by the procedures given in par. (a).

(c) The annual reciprocity fee shall be set to equal the minimum annual

certification fee.

Table 2 Fees for Certification and Registration

Item	Fee (Effective May 1994)
Base Fee	$250 per year
1. Oxygen Utilization	$ 25 per year
2. Nitrogen	$ 25 per year
3. Phosphorus	$ 25 per year
4. Physical	$ 25 per year
5. General I	$ 50 per year
6. General II	$ 50 per year
7. General III	$100 per year
8. Metals I	$100 per year
9. Metals II	$100 per year
10. Purgeable Organics	$100 per year
11. Base/Neutral Organics	$100 per year
12. Acid Organics	$100 per year
13. Liquid Chromatography	$100 per year
14. Acid Pesticides	$100 per year
15. Petroleum Hydrocarbons	$300 per year
16. Organochlorine Compounds	$100 per year
17. Dioxins	$300 per year
18. Safe Drinking Water	$500 per year
NO_3+NO_2 only	$ 50 per year
19. Any Single Analyte	$100 per year
20. Effluent Toxicity Testing	$650 per year
Reciprocity [1]	$600 per year
Initial Application	$150 for initial application
Revised Application	$ 75 per application
Late Renewal	$ 50
Evaluation of Out-of-State Labs	Additional Travel Costs
Enforcement Follow-up Evaluation	Actual Cost of Evaluation
Discretionary Acceptance	Actual Cost of Determining Data Quality

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

SECTION 6. NR 149.07 (1g) is created to read:

NR 149.07 (1g) If the laboratory has not submitted all the necessary materials described in sub. (3) (a) to (c), its application shall expire one year from the date the application is received by the department. If the laboratory then wishes to pursue certification or registration, the laboratory shall pay the appropriate fees and submit a new application per this section.

SECTION 7. NR 149.07(5) is created to read:

NR 149.07 (5) APPLICATION FOR TRANSFER OF LABORATORY CERTIFICATION OR REGISTRATION. (a) This subsection applies to a change in ownership that involves the purchase or lease of equipment and where greater than or equal to 60% of the analytical staff are retained. A change in ownership that involves the purchase or lease of equipment and where less than 60% of the analytical staff are retained shall be treated as an application under subs. (1) to (3).

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

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

2. Pay the revised application fee.

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

4. Agree to comply with this chapter.

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

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

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

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

SECTION 8. NR 149.13 (11) is amended to read:

NR 149.13 (11) For test category 17, no reference sample is required. The laboratory shall demonstrate, upon application for certification or

registration, acceptable precision and percent recovery based on ~~replicate analysis and spiked sample analysis~~ the successful completion of the initial precision and accuracy portion of the method. The department shall judge acceptability based on the criteria given in the method. ~~The following information shall be submitted:~~

~~(a) A detailed description of the methodology.~~

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

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

SECTION 9. NR 149.21 (Title) is amended to read:

NR 149.21 (Title) **Requirements For Safe Drinking Water Certification.**

SECTION 10. NR 149.21 (1) to (6) and (8) are renumbered to NR 149.21 (2) to (7) and (1) and 149.21 (1), (6) and (7), as renumbered, are amended to read:

NR 149.21 (1) GENERAL REQUIREMENTS FOR SAFE DRINKING WATER CERTIFICATION. (a) The criteria and procedures for safe drinking water certification are those criteria and procedures specified in "Manual for the Certification of Laboratories Analyzing Drinking Water", EPA/570/9-90/008, third edition, EPA, Office of Water, April 1990, including change 1, October 1991, and change 2, September 1992.

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

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

(6) REQUIREMENTS FOR INORGANIC CHEMICALS. To receive certification to conduct analyses for ~~asbestos, barium, cadmium, chromium, copper,~~ cyanide, fluoride, ~~lead, mercury~~ metals, nitrate, and 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~~ meet the acceptance limits~~,~~ listed in 40 CFR 141.23 (k)(6)(ii) and 141.89 (a)(1)(ii).

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

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

~~Note: Federal regulations include cis 1,2 dichloroethylene.~~

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

(b) Achieve quantitative results on the analyses performed under par. (a) 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~~ the acceptance limits listed in 40 CFR 141.24 (g)(11)(i)(C) and (D); and

~~(c) Achieve quantitative results on the analyses performed under par. (a) 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 volatile 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~~ 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~~.

SECTION 11. NR 149.21 (7) is repealed.

SECTION 12. NR 149.21 (8) is created to read:

NR 149.21 (8) REQUIREMENTS FOR OTHER ORGANIC COMPOUNDS. To receive certification to conduct analyses for synthetic organic contaminants and total trihalomethanes, the laboratory shall:

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

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

SECTION 13. NR 149.42 (1)(a)8 and (b)9 are created to read:

NR 149.42(1)(a)8. Conducting analysis for test categories for which certification has not been granted.

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

SECTION 14. NR 149.42 (1)(d)2 is amended to read:

NR 149.42 (1)(d)2. An order suspending or revoking a certification or revoking a registration shall take effect on the thirtieth day after the order is mailed, unless the owner of a certified or registered laboratory submits a ~~request~~ petition for a hearing to the department within 30 days. The ~~request~~ petition for hearing shall specify the findings or conclusions, or both, which the laboratory disputes and conform to the requirements of s. NR 2.05 (5). If a request is submitted and meets the requirements of s. 227.42, Stats., the suspension or revocation ~~is~~ shall be stayed and the department shall conduct a contested case hearing on the matter. At least 10 days prior to the date of the hearing, the department shall send a written notice to the laboratory indicating the date, time and location of the hearing. The final determination of the department, including the basis for the decision, shall be provided by written order to the laboratory after the hearing.

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

SECTION 15. NR 149.42 (2) is repealed and recreated to read:

NR 149.42 (2) REFERRAL. (a) Any violation of this chapter may be referred to the attorney general's office for enforcement under ss. 144.98 and 144.99, Stats.

(b) Any laboratory operating without proper certification or registration, for which analysis results are submitted to the department for compliance monitoring or for analyses which require certification or registration under ch. NR 605 or 630, may be referred by the department to the

attorney general's office for enforcement.

SECTION 16. NR 149.43 (title), (1) and (2) are repealed and recreated to read:

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

(2) PRIVATE ORGANIZATION AGREEMENTS. The department may recognize the certification, accreditation or approval of a laboratory by a private nonprofit organization if the organization's standards for certification,

accreditation or approval are substantially equivalent to those established under this chapter. The department may negotiate with and attempt to enter into acceptable agreements with private nonprofit organizations for the purpose of recognition under this subsection. The department shall publish periodically a list of those organizations whose certifications, accreditations or approvals it accepts. The department may accept the results of any tests conducted by a laboratory that it recognizes under an agreement. Any laboratory that is certified, accredited or approved by an organization with which the department has an agreement may apply to the department to be recognized under this subsection.

SECTION 17. NR 149.43 (3) (title) is created to read:

NR 149.43 (3) (title) HEALTH AND SOCIAL SERVICES AGREEMENT.

SECTION 18. NR 149.46 is repealed.

SECTION 19. NR 219.04 (1) is amended to read:

NR 219.04 (1) ANALYTICAL TEST PROCEDURES. Parameters or pollutants, for which wastewater analytical methods are approved, are listed together with test procedure descriptions and references in tables A to E. Parameters or pollutants, for which sludge analytical methods are approved, are listed together with test procedure descriptions and references in table EM. The discharge values for the listed parameters shall be determined by one of the standard analytical test procedures identified in a table under this

subsection or by an alternate test procedure established under ss. NR 219.05 and 219.06.

SECTION 20. NR 219.04 Table A, (title) and items 1 and 9 are amended to read:

TABLE A
LIST OF APPROVED BIOLOGICAL TEST PROCEDURES FOR WASTEWATER

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 ~~9222G~~ 9222D	B-0050-85[4]
Acute and Chronic Toxicity:				
9. Toxicity, acute, fresh water organisms, effluent[10]	Daphnia and Ceriodaphnia, 48-h static mortality.	p ~~39~~ 56 & 58[8]		
	Fathead minnow, 48-h static mortality, or 48 to 96-h flow-through mortality.	p ~~44~~ 60[8]		

SECTION 21. NR 219.04 Table B, (title) items 3, 5, 6, 7, 8, 9, 10, 12, 13, 18, 19, 20, 22, 26, 30, 32, 33, 34, 36, 37, 47, 51, 60, 62, 63, 72, 74, and 75 are amended to read:

NR 219.04

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

	Parameter, Units & Methods	EPA[1]	Standard Methods[3]	ASTM[3]	USGS[4]	Other
3.	Aluminum-Total[6], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	202.1	3111 D		I-3051-85	
	AA furnace,	202.2	3113 B			
	Inductively coupled plasma (ICP)[6m],	200.7[7]	3120 B			
	Direct current plasma (DCP)[6m], or			D4190-88		Note ~~34~~ 36
	Colorimetric (Eriochrome cyanine R)		3500-AlD			
5.	Antimony - Total[6], ug/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	204.1	3111 B			
	AA furnace, or	204.2	3113 B			
	Inductively coupled plasma[6m]	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[6m],	200.7[7]	3120 B			
	Or, colorimetric (SDDC)	206.4	3500-As	D2972-84(A)	I-3060-85	
7.	Barium-Total[6], mg/L:					

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

	Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
	Digestion[6] followed by:					
	AA direct aspiration[6m],	208.1	3111 D		I-3084-85	
	AA furnace,	208.2	3113 B			
	Inductively coupled plasma[6m], or	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6n]					Note ~~34~~ 36
8.	Beryllium-Total[5], 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~~ Direct current plasma, or			D4190-88		Note ~~34~~ 36
	Colorimetric (aluminon)		3500-Be D			
9.	Biochemical oxygen demand (BOD5), mg/L:					
	Winkler (Azide modifications)	~~405.1~~	5210		I-1578-78[10]	973.443[3] 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~~ Direct current plasma			D4190-88		Note ~~34~~ 36
12.	Cadmium-Total[5], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	213.1	3111 B or C	D3557-90(A or B)	I-3135-85 or I-3136-85	974.27[3] p.37[11]
	AA furnace,	213.2	3113B			
	Inductively coupled plasma[6m]	200.7[7]	3120B		I-1472-85	
	~~DCP~~ Direct current plasma[6n],			D4190-90		Note ~~34~~ 36
	Voltametry[13], or			D3557-90(C)		
	Colorimetric (Dithizone)		3500-Cd D			
13.	Calcium-Total[5], 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~~ Direct current plasma, or					Note ~~34~~ 36
	EDTA titration	215.2	3500-Ca D	D511-88(A)		
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), or				I-1230-85	307B[19]
	Ion Chromatography					218.6[19n]
19.	Chromium-Total[5], mg/L:					
	Digestion[6] (optional extraction) followed by:					
	AA direct aspiration[6m],	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[6m],	200.7[7]	3120B			
	~~DCP~~ Direct current plasma[6n], or			D4190-88		Note ~~34~~ 36
	Colorimetric (diphenylcarbazide),		3500-Cr D	D1687-84(A)		
20.	Cobalt-Total[5], 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~~ Direct current plasma			D4190-88		Note ~~34~~ 36
22.	Copper-Total[5], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	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			

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

	Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
	Inductively coupled plasma[6m]	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6m],			D4190-88		Note ~~34~~ 36
	Colorimetric (Neocuproine), or		3500-Cu D or E	D1688-84(88)(A)		
	Bicinchoninate					Note 21
26.	Gold Total[8], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration	231.1	3111 B			
	AA furnace, or	231.2	3113 B			
	~~DCP~~ Direct current plasma					Note ~~34~~ 36
30.	Iron-Total[8], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	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[6m],	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6m], or					Note ~~34~~ 36
	Colorimetric (Phenanthroline)		3500-Fe D	D1068-84(A)		Note 24
32.	Lead-Total[8], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	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[6m],	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6m],			D4190-88		Note ~~34~~ 36
	Voltametry[13] or			D3559-90(C)		
	Colorimetric (Dithizone)		3500-Pb D			
33.	Magnesium-Total[8], 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~~ Direct current plasma, or					Note ~~34~~ 36
	Gravimetric		3500-Mg D	D511-77(A)		
34.	Manganese-Total[8], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	243.1	3111 B or C	D858-90	I-3454-85	974.27[5]
	AA furnace,	243.2	3113 B	(A or B)		
	Inductively coupled plasma[6m],	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6m],			D4190-88		Note ~~34~~ 36
	Colorimetric (Persulfate), or		3500-Mn D	D858-84(A)(1988)		920.203[5]
	Periodate					Note 25
36.	Molybdenum-Total[8], 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~~ Direct current plasma					Note ~~34~~ 36
37.	Nickel-Total[8], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	249.1	3111 B or C	D1886-90 (A or B)	I-3499-85	
	AA furnace,	249.2	3113 B			
	Inductively coupled plasma[6m],	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6m], or			D4190-88		Note ~~34~~ 36
	Colorimetric (Heptoxime)		3500-Ni D			
47.	Palladium-Total[8], 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~~ Direct current plasma					Note ~~34~~ 36
51.	Platinum-Total[8], mg/L:					
	Digestion[6] followed by:					

TABLE B (continued)

LIST OF APPROVED INORGANIC TEST PROCEDURES

	Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
	AA direct aspiration,	255.1	3111 B			
	AA furnace, or	255.2				
	~~DCP~~ Direct current plasma					Note ~~34~~ 36
60.	Selenium - Total[6] ug/L:					
	Digestion[6] followed by:					
	AA furnace,	270.2	3113 B			
	Inductively coupled plasma[6m],	200.7[7]	3120 B			
	or AA (gaseous hydride)		3114 B	D3859-88(A)	I-3667-85	
62.	Silver-Total[11], mg/L:					
	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~~ Direct current plasma					Note ~~34~~ 36
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~~ Direct current plasma, or					Note ~~34~~ 36
	Flame photometric		3500-Na D	D1428-82(A)		
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~~ Direct current plasma					Note ~~34~~ 36
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~~ Direct current plasma, or			D4190-88		Note ~~34~~ 36
	Colorimetric (Gallic acid)		3500-V D	D3373-84(A) (1988)		
75.	Zinc-Total[6], mg/L:					
	Digestion[6] followed by:					
	AA direct aspiration[6m],	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[6m],	200.7[7]	3120 B			
	~~DCP~~ Direct current plasma[6m],			D4190-88		Note ~~34~~ 36
	Colorimetric (Dithizone), or		3500-Zn E			
	Colorimetric (Zincon)		3500-Zn F			Note ~~34~~ 36

SECTION 22. NR 219.04, Table B, Note [6] is repealed and recreated to read:

NR 219.04, Table B, Note [6] For the determination of total metals and total recoverable metals the sample is not filtered before processing. Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. 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 and 1983. The total metals digestion is a vigorous digestion using nitric acid (4.1.3). The total recoverable metals digestion is a less vigorous digestion using nitric and hydrochloric acids (4.1.4). Use of the graphite furnace technique, inductively coupled plasma, direct current plasma, as well as determinations for certain elements such as arsenic, mercury, selenium, silver, and titanium require a modified digestion and in all cases the method should be consulted for specific instructions and/or cautions. If the digestion included in one of the other approved references is different than the above, the EPA procedure shall be used.

Sample digestion may be omitted for AA (direct aspiration or graphite furnace), direct current plasma, and inductively coupled plasma 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.

SECTION 23. NR 219.04, Table B, Notes [6m] and [19m] are created to read:

NR 219.04, Table B, Note [6m] "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106-0200, April 16, 1992. Available from the CEM Corporation.

[19m] "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Office of Research and Development, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91-231498, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

SECTION 24. NR 219.04, Table B, Note [7] and [36] are amended to read:

NR 219.04, Table B, Note [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~~ 20402.

[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~~ Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.

SECTION 25. NR 219.04 Table C, (title) is amended to read:

TABLE C
LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS IN WASTEWATER

SECTION 26. NR 219.04 Table D, (title) is amended to read:

TABLE D
LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES[1] IN WASTEWATER

SECTION 27. NR 219.04 Table E, (title) is amended to read:

TABLE E
LIST OF APPROVED RADIOLOGICAL TEST PROCEDURES FOR WASTEWATER

SECTION 28. NR 219.04, Table EM is created to read:

TABLE EM
APPROVED ANALYTICAL METHODS FOR SLUDGE

Parameter	Digestion	Method	Method Number
Metals[1]			
Arsenic	3050A	Inductively Coupled Plasma Emission	6010A
Arsenic	7061A	Gaseous Hydride[2]	7061A
Arsenic	3050A	Graphite Furnace	7060A
Beryllium	3050A	Inductively Coupled Plasma Emission	6010A
Beryllium	3050A	Flame Atomic Absorption	7090
Beryllium	3050A	Graphite Furnace	7091

Parameter	Digestion	Method	Method Number
Cadmium	3050A	Inductively Coupled Plasma Emission	6010A
Cadmium	3050A	Flame Atomic Absorption	7130
Cadmium	3050A	Graphite Furnace	7131A
Chromium	3050A	Inductively Coupled Plasma Emission	6010A
Chromium	3050A	Flame Atomic Absorption	7190
Chromium	3050A	Graphite Furnace	7191
Copper	3050A	Inductively Coupled Plasma Emission	6010A
Copper	3050A	Flame Atomic Absorption	7210
Lead	3050A	Inductively Coupled Plasma Emission	6010A
Lead	3050A	Flame Atomic Absorption	7420
Lead	3050A	Graphite Furnace [3]	7421
Mercury	7471A	Cold Vapor	7471A
Molybdenum	3050A	Inductively Coupled Plasma Emission	6010A
Molybdenum	3050A	Flame Atomic Absorption	7480
Molybdenum	3050A	Graphite Furnace	7481
Nickel	3050A	Inductively Coupled Plasma Emission	6010A
Nickel	3050A	Flame Atomic Absorption	7520
Selenium	3050A	Inductively Coupled Plasma Emission	6010A
Selenium	7741A	Gaseous Hydride [2]	7741A
Selenium	3050A	Graphite Furnace	7740
Zinc	3050A	Inductively Coupled Plasma Emission	6010A
Zinc	3050A	Flame Atomic Absorption	7950
Biological			
Enteric viruses	NA	Centrifuge Concentration	D 4994-89[4]
Fecal coliform	NA NA	Most Probable Number Membrane Filter	9221 E or 9222 D [5]
Helminth ova	NA	Density Gradient Floatation	[6]
Specific Oxygen Uptake Rate	NA	Respirometer	2710 B [5]
Salmonella	NA	Most Probable Number Selective Media Culture	9260 D.1 [5] [7]

Parameter	Digestion	Method	Method Number
Physical			
Solids	NA	Gravimetric	2540 G [5]
Percent Volatiles Solids Reduction	NA	Calculation	[8]

TABLE EM NOTES

[1] "Test Methods for Evaluating Solid Waste", SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987 and July 1992 updates, Washington, DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250-7954, (202) 783-3238.

[2] High levels of chromium, copper, mercury, silver, cobalt, or molybdenum may interfere with the analysis. Consult method 3114, of "Standard Method for the Examination of Water and Wastewater", 17th or 18th edition, for more information.

[3] Concentrations of lead in municipal sludge may exceed the working range of Graphite Furnace.

[4] "1991 Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[5] "Standard Methods for the Examination of Water and Wastewater", 18th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[6] "Occurrence of Pathogens in Distribution and Marketing Municipal Sludges", EPA 600/1-87-014, Environmental Protection Agency, 1987. Available from the National Technical Information Service, order # PB 88-154273/AS, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

[7] "Determination and Enumeration of Salmonella and Pseudomonas aeruginosa", Kenner, B.A. and H.A. Clark, J. Water Pollution Control Federation, 46(9):2163-2171, 1994. Available from the Water Environment Federation, 601 Wythe St., Alexandria, VA 22314.

[8] "Environmental Regulations and Technology - Control of Pathogens and Bextors in Sewage Sludge", EPA-625/R-92/013, Environmental Protection Agency, Cincinnati, OH, 1992. Available from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

SECTION 29. NR 219.04, Table F, (title) is amended to read:

TABLE F
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR WASTEWATER

SECTION 30. NR 605.08 (5)(a) is amended to read:

NR 605.08(5) TOXICITY CHARACTERISTIC. (a) A solid waste exhibits the characteristic of toxicity if, using the ~~test methods described in 40 CFR 261, Appendix II, July 1, 1990, or equivalent methods approved by EPA under the procedures set forth in 40 CFR 260.20 and 260.21~~ toxicity characteristic leaching procedure, EPA method 1311 in SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", third edition, September, 1986, as amended by update I in July, 1992, the extract from a representative sample of the waste contains any of the contaminants listed in table I at a concentration equal to or greater

than the respective value given in that table. Where the waste contains less than 0.5% filterable solids, the waste itself, after filtering using the methodology in ~~40 CFR 261, Appendix II, July 1, 1990~~ method 1311, is considered to be the extract for the purpose of this ~~section~~ subsection.

Note: ~~The publication containing the CFR references~~ Publication SW-846 may be obtained from:

~~The~~ Superintendent of Documents
U.S. Government Printing Office
~~Washington, D.D. 20402~~
P.O. Box 371954
Pittsburgh, PA 15250-7954
(202) 783-3238

This publication is available for inspection at the offices of the department, the secretary of state and the revisor of statutes.

The foregoing rule was approved and adopted by the State of Wisconsin Natural Resources Board on December 9, 1993.

The rule 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.

Dated at Madison, Wisconsin March 3, 1994

STATE OF WISCONSIN
DEPARTMENT OF NATURAL RESOURCES

By George E. Meyer
George E. Meyer, Secretary

(SEAL)

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REVISOR OF STATUTES BUREAU