95-048

WISCONSIN
DEPT. OF NATURAL RESOURCES
George E. Meyer
Secretary

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

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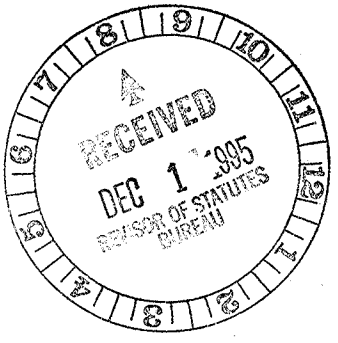
STATE OF WISCONSIN)
) ss
DEPARTMENT OF NATURAL RESOURCES)

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-22-95 was duly approved and adopted by this Department on September 28, 1995. 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 22nd day of November, 1995

George E. Meyer

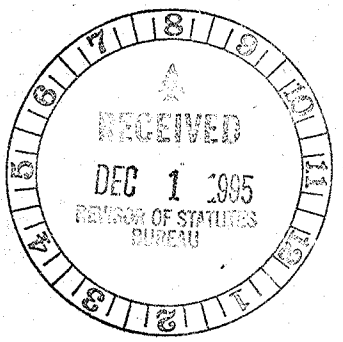
George E. Meyer, Secretary

(SEAL)

2-1-96

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

The Wisconsin Natural Resources Board proposes an order to repeal ss. NR 149.05(5); to renumber NR 149.07(1g) to (5) to 149.07(2) to (7); to renumber and amend NR 149.12 to 149.12(1); to amend NR 149.02(note), 149.03(5)(c), 149.03(17), (29) & 32, 149.04(1) Table 1 (items 2, 10 & 13), 149.06(1)(intro.), 149.11(1)(note) & (3), 149.14(3)(c)4, 149.14(3)(e) & (f)(intro.), 149.21(7)(b), 149.44, 219.04(1) & (2), 219.04 Table A (title), (header) and (items 1 & 2), 219.04 Table E (note 2) and (note 3), 219.04 table EM (header) and (note 4), 219.05, and 219.06; to repeal & recreate ss. NR 149.04(1) Table 1 (items 11, 12, note & 14), 149.05(1) & Table 2, and NR 219.04 Tables B, C, D, & F; and to create ss. NR 149.02(4), 149.03(5)(n), (o), (p), & (q), 149.03(24m), (note), (28m) & (31m), 149.07(1)(g), (note) & (6)(c), 149.11(4), 149.12(2) & note, 149.15, 149.21(9), NR 219.04 table BM, 219.04 table EM (note 19), and NR 700.13 & (note) of the Wisconsin Administratitive Code relating to laboratory certification and registration, sample preservation procedures, analytical methodology, and laboratory procedures.



Analysis Prepared by the Department of Natural Resources

Statutory Authority: ss. 144.431, 144.62, 144.95, 147.08, 159.03 and 227.11, Stats.

Statutes Interpreted: ss. 144.431, 144.62, 144.95, 147.08 and 159.03 Stats.

The effect of amending ch. NR 149, Wis. Adm. Code, is to: 1) Clarify the records retention provision and add a provision requiring laboratories to retain analytical records generated during the certification period for 3 years [SECTIONs 1 & 12], 2) List department programs which require a certified laboratory to perform analytical testing [SECTION 2], 3) Update reference to EPA SW-846 authoritative source [SECTION 3], 4) Add the Wisconsin GRO method, Wisconsin DRO method, EPA's Technical Notes on Drinking Water and U.S. Army Toxic and Hazardous Materials Quality Assurance Program as "authoritative sources" [SECTION 4], 5) Clarify when the sample is spiked for the "method of standard addition" versus a "matrix spike" and add a provision allowing methanol trip blanks[SECTIONs 5 & 21], 6) Add definitions for "received on ice", "sensitivity" and "temperature blank" [SECTION 6], 7) Add inorganic nitrate + nitrite, aldehydes and ketones, carbamate pesticides and other pesticides by LC as certifiable parameters [SECTION 7], 8) Divide semivolatile parameters into two separate test categories for semivolatiles by gas chromatography or semivolatiles by gas chromatography/mass spectroscopy and consolidate pesticides subcategories [SECTION 8], 9) Implement a formula for calculating fee changes for the program [SECTIONs 9 & 10], 10) Repeal s. NR 149.05(5), the provision allowing prorated fees [SECTION 11], 11) Add a provision allowing the department to return incomplete applications to the applicants [SECTIONs 13 & 14], 13) Add a provision requiring method detection limit studies, calibration information or other information to be submitted with an application for certain parameters [SECTION 15], 14) Add a provision clarifying when certification expires if a lab does not renew annually [SECTION 15], 15) Clarify the proper sample preservation procedures and holding times [SECTION 16], 16) Add a provision for clarifying sample temperature and holding time exceedances reporting [SECTIONs 17 & 22], 17) Clarify EPA approval of alternate methodology [SECTION 18], 18) Add a provision allowing the department to approve the use of emerging technology methods based upon current statutory authority [SECTION 19], 19) Specify the required known standard for biochemical oxygen demand analysis [SECTION 20], 20) Change the requirement for analyzing replicate samples [SECTION 21], 21) Clarify the use of matrix spikes (spiked samples) [SECTION 21], 22) Add a provision for data reporting procedures, including the identity of subcontracted labs [SECTION 22], 23) Add a provision requiring reporting all analytical results greater than the limit of detection [SECTION 22], 24) Update federal code citations [SECTIONs 16 & 23], 25) Add a provision requiring drinking water labs to notify the facility within 48 hours when

samples exceed the maximum contaminant levels for any regulated analyte [SECTION 24] and 26) Clarify the intent of discretionary data acceptance [SECTION 25].

The effect of amending ch. NR 219, Wis. Adm. Code is to: 1) Update code citations [SECTION 26], 2) Add reference to digestion table [SECTION 26], 3) Update method references in the tables, including the 18th edition of "Standard Methods for the Examination of Water and Wastewaster" [SECTIONs 28, 29, 31, 32, 33 & 34], 4) Clarify sample temperature reporting and variances for consistency with NR 149 [SECTION 27], 5) Incorporate changes to the federal rules on pages 4504 to 4515 of the January 31, 1994 Federal Register pertaining to analysis of wastewater effluent samples [SECTIONs 28, 29, 31, 32, 34 & 36], and on pages 62456 to 62471 of the December 5, 1994 Federal Register [SECTION 29], 6) Incorporate EPA solid waste methods (SW-846) into the approved methodology [SECTIONs 29, 30, 31 & 32], 7) Add organic mercury methods to inorganics table and add EPA approved methods 200.7 (ICP), 200.8 (ICP/MS), 200.9 (STGFAA), 300.0 (ion chromatography) and semi-automated colorimetric methods 335.4, 420.4, and 375.2 to applicable parameters [SECTION 29], 8) Delete reference to "American National Standard on Photographic Processing Effluents" [SECTION 29], 9) Add "Standard Methods" 5520 C & D for chemical oxygen demand and 5310-B,C & D for total organic carbon [SECTION 29], 10) Clarify and compare approved metals sample preparatory procedures [SECTION 30], 11) Rearrange non-pesticide organics table for clarity [SECTION 31], 12) Delete choroethylvinyl ether from organics table [SECTION 31], 13) Add "Standard Methods" 6210 B" for acrolein and acrylonitrile [SECTION 31], 14) Separate packed column and capillary column GC SW-846 methods [SECTIONs 31 & 32], 15) Clarify proper sludge digestion procedure for metals analysis [SECTIONs 30 & 34], 16) Clarify approval of alternate test procedures [SECTION 37] and 17) Correct miscellaneous editorial errors [SECTIONs 29, 31, 32 & 36].

The effect of amending ch. NR 700, Wis. Adm. Code is to 1) Require methanol preservation for soil samples collected for purgeable organics analysis and to specify the maximum holding times for solvent addition and analysis of samples collected for compliance with chs. NR 700 to 736. [SECTION 38].

SECTION 1. NR 149.02(4) is created to read:

NR 149.02(4) Section NR 149.06 applies to the custodians of the records of any of the following:

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

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

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

SECTION 2. NR 149.02 (note) is amended to read:

Note: Administrative Codes and Programs requiring analyses to be done by a certified or registered laboratory are chs. NR 110- Sewerage Systems, 113- Servicing Septic Systems, 123- Well Compensation Program, 131- Metallic Mineral Prospecting, 132- Metallic Mineral Mining, 140- Groundwater Quality, 145- Private Wells, 150- Environmental Analysis and Review Procedures, 157- Management of PCBs, 158- Hazardous Substance Discharge Notification, 182- Metallic Mining Waste, 210- Sewage Treatment Works, 211- General Pretreatment Requirements, 212- Wasteload Allocated Effluent Limits, 219- Analytical Test Methods, 347- Sediment Sampling and Analysis, 508- Landfill Monitoring and Remedial Actions, 605- Identification of Hazardous Waste, 630- Storage, Treatment, and Disposal Facilities, 716- Site Investigation and 809- Safe Drinking Water.

SECTION 3. 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~~, July 1992, September 1994 and January 1995 updates.

SECTION 4. NR 149.03(5)(n), (o), (p) and (q) are created to read:

NR 149.03(5)(n) "Modified GRO- Method for Determining Gasoline Range Organics", WI-PUBL-SW-140, Wisconsin Department of Natural Resources, 101 S. Webster St., Madison, WI, 53707, September 1995.

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

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

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

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

SECTION 5. NR 149.03(17), (29) and (32) are amended to read:

NR 149.03(17) "Method of standard addition" means an analytical technique used to quantify samples whose matrices differ significantly from those of the known standards, ~~which~~and is accomplished by analyzing the sample and mixtures of the sample with at least 3 known standards, and either plotting the analytical response versus the added concentration and extrapolating the plot to determine the original concentration of the analyte in the sample or by calculating the analytical response for the analyte based upon a least squared regression to determine the original concentration of the analyte in the sample. The samples are processed through all preparative steps prior to the standard additions.

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

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

SECTION 6. NR 149.03(24m), (note), (28m) and (31m) are created to read:

NR 149.03(24m) "Received on ice" means that sample containers are surrounded by an ice slurry,. or crushed, cubed or chipped ice at the time of receipt in the laboratory.

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

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

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

SECTION 7. NR 149.04(1) Table 1 items 2, 10 and 13 are amended to read:

2.	Nitrogen	Each analyte for which certification or registration is desired except nitrite.	Nitrate as Nitrogen, Nitrite as Nitrogen, Ammonia as Nitrogen, total Kjeldahl Nitrogen, Nitrate + Nitrite as Nitrogen.
10.	Organics; Purgeable by Gas Chromatography or Gas Chromatography/Mass Spectrometry	Representative purgeable analytes.	Purgeable Halocarbons, Purgeable Aromatics, ~~Arolein~~Acrolein, Acrylonitrile.
13.	Organics; Extractables by Liquid Chromatography	Representative Polynuclear Aromatic Hydrocarbons, Benzidines, ~~or~~and Pesticides analyzable by liquid chromatography.	Benzidines, Polynuclear Aromatic Hydrocarbons, Aldehydes and Ketones, Carbamate Pesticides subject to Liquid Chromatography (e.g., carbofuran, oxamyl, and methomyl) and Other Pesticides subject to Liquid Chromatography (e.g., diquat and paraquat).

SECTION 8. NR 149.04(1) Table 1 items 11, 12, (note) and 14 are repealed and recreated to read:

11.	Organics; Semivolatiles by Gas Chromatography	Representative analytes within each subcategory for which certification is desired. The following subcategories are included: Phenolic Compounds (acid-extractables), Phthalate Esters, Nitrosamines, Nitroaromatics and Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons.	Phenolic Compounds (acid-extractables), Phthalate Esters, Nitrosamines, Nitroaromatics and Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons.
12.	Organics; Semivolatiles by Gas Chromatography/Mass Spectrometry	Representative analytes within each subcategory for which certification is desired including Phenolic Compounds. **(Note: All semivolatiles included in a particular study must be analyzed and reported. To be considered a representative sample for base/neutral extractables, a study must include at least 4 subcategories of base/neutral extractables.)**	Phenolic Compounds (acid-extractables) and Base/Neutral Extractable Compounds (excluding pesticides).
14.	Organics; Pesticides	Representative pesticides within each subcategory for which certification is desired.	Acid Herbicides (e.g., 2,4-D, 2,4,5-T, picloram, etc.), Nitrogen Pesticides, Organophophorus Pesticides, Triazine Pesticides (including metabolites) and Other Pesticides.

SECTION 9. NR 149.05(1) and Table 2 are repealed and recreated to read:

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

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

1. Fee Income $\leq$ ASA - TR

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

b. TR is the total out-of-state travel reimbursements in a given fiscal year.

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

d. Estimates of the fee income and travel reimbursement shall be calculated according to par. (c).

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

2. Total # RV Units = $\Sigma_{items1\text{-}26}$ (# Labs in Item)(RV of Item)

a. Total # RV units is the total number of relative value (RV) units available for the fiscal year. The relative value of each fee item (RV of item) is listed in table 2.

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

c. Total # of RV units is calculated by summing the product of (RV of item) and (# labs in item) for each individual item.

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

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

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

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

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

Table 2
Fees for Certification and Registration

Item	Relative Value[1]
1. Base Fee	10
2. Cat. 1 - Oxygen Utilization	1
3. Cat. 2 - Nitrogen	1
4. Cat. 3 - Phosphorus	1

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

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

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

SECTION 10. NR 149.05(4) is repealed and recreated to read:

NR 149.05(4) FEE REVISION. The department may amend the formulas in this section based upon a demonstrated need for revision to support the level of effort in the program. Any amendments to the formulas in this

section shall be reviewed by the laboratory certification standards review council prior to being proposed as rule amendments.

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

SECTION 11. NR 149.05(5) is repealed.

SECTION 12. NR 149.06(1)(intro.) is amended to read:

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

SECTION 13. NR 149.07(1)(a) is amended to read:

NR 149.07(1)(a) Complete an application and submit it with the appropriate fees prescribed in s. NR 149.05. Incomplete applications and applications received without the appropriate fees may be returned to the applicant unprocessed if any of the information required in this subsection is not included with the application.

SECTION 14. NR 149.07(1g) to (5) are renumbered to NR 149.07(2) to (7).

SECTION 15. NR 149.07(1)(g), (note) and (6)(c) are created to read:

NR 149.07(1)(g) Submit other analyte specific information as required by the method or the department.

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

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

SECTION 16. NR 149.11(1)(note) and (3) are amended to read:

Note: Analytical methodologies required by state regulations are in chs. NR ~~809,~~ 219, 508, ~~and~~ 605, 675, 700 and 809. Thoe required by federal regulations are in 40 CFR 136, 141 and ~~261~~268.

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

SECTION 17. NR 149.11(4) is created to read:

NR 149.11(4) Samples requiring preservation at 4 °C under this section may be recorded as "received on ice" only if solid ice is present in the cooler at the time the samples are received. Samples cooled during shipping with ice packs may not be recorded as received on ice. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

(a) The temperature of an actual sample.

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

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

SECTION 18. NR 149.12 is renumbered NR 149.12(1) and amended to read:

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

SECTION 19. NR 149.12(2) and (note) are created to read:

NR 149.12(2) EMERGING TECHNOLOGY. The department may allow alternate methods which use existing new or innovative technologies on a case-by-case basis. Laboratories may request approval for an emerging technology method by following the 2 step approval process outlined in pars. (a) and (b). Modifications to an approved method may not be considered an emerging technology. Laboratories shall request approval for modifications to an approved method according to sub. (1).

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

(b) The department may approve or deny the request within 90 days based on a demonstrated department need for the emerging technology method. If the request is granted, the department will establish criteria for validating the method on a case-by-case basis. If the method validation meets the predetermined criteria, the department shall permit the use of the method. The department may charge a fee under s. 144.95(5)(d), Stats., if it is necessary to verify the results of the data.

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

SECTION 20. NR 149.14(3)(c)4 is amended to read:

NR 149.14(3)(c)4 For test category 1, a known standard shall be analyzed after the analysis of 20 samples or once a week. The known standard for biochemical oxygen demand shall be glucose/glutamic acid. The limits on this quality control check shall be as established in an authoritative source or those established by the provider, whichever is more stringent.

SECTION 21. NR 149.14(3)(e) and (f)(intro.) are amended to read:

NR 149.14(3)(e) A replicate sample shall be run after the analysis of ~~10~~ 20 samples for each matrix type, unless the methodology specifies otherwise. No replicate samples are needed for oil and grease.

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

SECTION 22. NR 149.15 and note are created to read:

NR 149.15 DATA REPORTING With each set of sample results, a laboratory shall report:

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

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

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

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

SECTION 23. NR 149.21(7)(b) is amended to read:

NR 149.21(7)(b) Achieve quantitative results on the analyses performed under par. (a) that are within the acceptance limits listed in ~~40 CFR 141.24 (g)(11)(i)(C) and (D)~~40 CFR 141.24(f)(17)(i)(C) and (D) and 40 CFR 141.24(f)(17)(ii)(B); and

SECTION 24. NR 149.21(9) is created to read:

NR 149.21(9) NOTIFICATION. A laboratory certified under this chapter for category 18, safe drinking water, and analyzing a drinking water sample for analytes regulated under ch. NR 809, shall notify the facility immediately but no later than 48 hours if a test shows that a compliance sample exceeds the MCL for any regulated analyte.

SECTION 25. NR 149.44 is amended to read:

NR 149.44 DISCRETIONARY ACCEPTANCE. The department may accept on a case-by-case basis the results of a test in a specified test category even though the test was not conducted by a certified or registered laboratory. The department may charge a fee under s. 144.95(5)(d), Stats., if it is necessary to verify the results of a test submitted under this section. The department may not accept data that do not meet the requirements established in this chapter. This section does not apply to monitoring required under ch. NR 809, where a certified laboratory is required.

SECTION 26. NR 219.04(1) and (2) are 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. Metals samples digestion procedures and references are listed in table BM. 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 allowed under ss. NR 219.05 and ~~219.06~~149.12.

(2) SAMPLE 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 where required. All samples requiring preservation at 4 °C shall be cooled immediately after collection, and the required temperature maintained during shipping. 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 shall make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

SECTION 27. NR 219.04(3) is created to read:

NR 219.04(3) TEMPERATURE REPORTING PROCEDURES. Samples cooled with ice packs or not in direct contact with ice during shipping shall be cooled to 4° C prior to shipping, and a temperature blank shall be submitted with the samples. Samples cooled during shipping with ice packs may not be recorded as received on ice. Samples may be recorded as received on ice only if solid ice is present in the cooler at the time the samples are received. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

(a) The temperature of an actual sample.

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

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

Note: Copies of the publications referenced in Tables A - F are available for inspection at the offices of the department of natural resources, the secretary of state and the revisor of statutes. Many of these materials are also available through inter-library loan.

SECTION 28. NR 219.04 Table A title, header and items 1 and 2 are amended to read:

TABLE A

LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter and Units	Method[1]	EPA	Standard Methods ~~17th~~18th Ed.	USGS
BACTERIA:				
1. Coliform (fecal) number per 100 ml	MPN, 5 tube dilution; or, membrane filter (MF)[2], single step	p132[3] p124[3]	~~9221C~~9221E 9222D	B-0050-85[4]
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube dilution; or, MF, single step[5]	p132[3] p124[3]	~~9221C~~9221E 922D	

SECTION 29. NR 219.04 Table B is repealed and recreated to read:

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
1. Acidity, as $CaCO_3$, mg/L,	305.1		2310 B(4a)	D1067-92		

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
Electrometric end point or phenolphthalein end point						
2. Alkalinity, as $CaCO_3$, mg/L; Electrometric or colorimetric:						
Titration to pH 4.5, manual	310.1		2320 B	D1067-92	I-1030-85	973.43[5]
Or automated	310.2					
3. Aluminum, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	202.1	7020	3111 D		I-305I-85	
AA furnace,	202.2 or 200.9[1g]		3113 B			
Inductively coupled plasma (ICP)[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry (ICP-MS),	200.8[1g]	6020				
Direct current plasma (DCP)[6m], or				D4190-82(88)		Note 36
Colorimetric (Eriochrome cyanine R)			3500-Al D			
4. Ammonia (as N), mg/L: Manual						
distillation[8] (at pH 9.5):	350.2		4500-NH_3 B			
Followed by						973.49[5]
Nesslerization,	350.2		4500-NH_3 C	D1426-89(A)	I-3520-85	973.46[5]
Titration,	350.2		4500-NH_3 E			
Electrode,	350.3		4500-NH_3 F & G	D1426-89(B)		
Automated phenate, or	350.1[1m]		4500-NH_3 H		I-4523.85	
Automated electrode						Note 9
5. Antimony, ug/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	204.1	7040	3111 B			
AA furnace,	200.9[1g]	7041	3113 B			
AA (gaseous borohydride),		7062				
Inductively coupled plasma[6m], or	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
6. Arsenic, ug/L:						
Digestion[6] followed by	206.5					
AA (gaseous hydride),		7061A	3114 B[37]	D2972-88(B)	I-3062.85	
AA (gaseous borohydride),		7062				
AA furnace,	206.2 or 200.9[1g]	7060A	3113 B	D2972-88(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
Or, colorimetric (SDDC)			3500-As C	D2972-88(A)	I-3060-85	
7. Barium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	208.1	7080A	3111 D		I-3084-85	
AA furnace,	208.2	7081	3113 B	D4382-91		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry, or	200.8[1g]	6020				
Direct current plasma[6m]						Note 36
8. Beryllium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	210.1	7090	3111 D	D3654-(88)(A)	I-3095-85	
AA furnace,	210.2, or 200.9[1g]	7091	3113 B	D3645(88)(B)		
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
Direct current plasma, or				D4190-82(88)		Note 36
Colorimetric (aluminon)			3500-Be D			
9. Biochemical oxygen demand (BOD_5),						

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

	Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
	mg/L:						
	Dissolved Oxygen Depletion			5210 B		I-1578-78[10]	973.443[5]
10.	Boron, mg/L:						
	Colorimetric (curcumin),	212.3		4500-B B		I-3112-85	
	Inductively coupled plasma, or	200.7[1g]	6010A	3120 B			
	Direct current plasma				D4190-82(88)		Note 36
11.	Bromide, mg/L: Titrimetric	320.1			D1246-82(88) (C)	I-1125-85	p.S44[12]
	Ion Chromatography	300.0[1m]	9056				
12.	Cadmium-Total[6], mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration6m,	213.1	7130	3111 B or C	D3557-90 (A or B)	I-3135-85 or I-3136-85	974.27[5]
	AA furnace,	213.2, or 200.9[1g]	7131A	3113 B	D3557-90(D)		
	Inductively coupled plasma[6m]	200.7[1g]	6010A	3120 B		I-1472-85	
	Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
	Direct current plasma[6m],				D4190-82(88)		Note 36
	Voltametry[13], or				D3557-90(C)		
	Colorimetric (Dithizone)			3500-Cd D			
13.	Calcium, mg/L:						
	Digestion[6] followed by:						
	Atomic absorption,	215.1	7140	3111 B	D511-92(B)	I-3152-85	
	Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
	Direct current plasma, or						Note 36
	EDTA titration	215.2		3500-Ca D	D511-92(A)		
14.	Carbonaceous Biochemical oxygen demand (CBOD5), mg/L: with nitrification inhibitor[14]			5210 B			
15.	Chemical oxygen demand (COD), mg/L:						
	Closed reflux			5220 C or D			Notes 15 & 16
	Titrimetric						
		410.1		5220 B	D1252-88(A)	I-3560 or I-3562-85	973.46[5]
		410.2					
		410.3					
	Automated and manual	410.4[1m]				I-3561-85	
	Spectrophotometric				D1252-88(B)		
16.	Chloride, mg/L:						
	Titrimetric (silver nitrate) or		9253	4500-Cl^- B	D512-89(B)	I-1183-85	
	(Mercuric nitrate),	325.3	9252A	4500-Cl^- C	D512-89(A)	I-1184-85	973.51[5]
	Colorimetric (ferricyanide), manual					I-1187-85	
	or automated, or	325.1 or 325.2	9250	4500-Cl^- E		I-2187-85	
	Ion chromatography	300.0[1m]	9056				
17.	Chlorine - Total residual, mg/L:						
	amperometric,	330.1		4500-Cl D	D1253-86(92)		
	Starch End point direct	330.3		4500-Cl B			
	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			4500-Cl I			Note 18
18.	Chromium VI dissolved, ug/L: 0.45 micron filtration with:						
	Extraction and atomic absorption,	218.4	7197	3111 A		I-1232-85	

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

	Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
	Coprecipitation and atomic absorption,		7195				
	Differential pulse polarography,		7198				
	Colorimetric (Diphenylcarbazide), or		7196A	3500-Cr D	D1687-92(A)	I-1230-85	307B[19]
	Ion Chromatography	218.6[1g]					
19.	Chromium, mg/L:						
	Digestion[6] (optional extraction) followed by:						
	AA direct aspiration[6m],	218.1	7190	3111 B	D1687-92(B)	I-3236-85	974.24[5]
	AA chelation extraction,	218.3		3111 C			
	AA furnace,	218.2, or 200.9[1g]	7191	3113B	D1687-92(C)		
	Inductively coupled plasma[6m],	200.7[1g]	6010A	3120B			
	Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
	Direct current plasma[6m], or				D4190-82(88)		Note 36
	Colorimetric (diphenylcarbazide),			3500-Cr D			
20.	Cobalt, mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration,	219.1	7200	3111 B (A or B)	D3558-90(A or B)	I-3239-84	
	AA furnace, or	219.2, or 200.9[1g]	7201	3113 B	D3558-90(C)		
	Inductively coupled plasma, or	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
	Direct current plasma				D4190-82(88)		Note 36
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, mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration6m,	220.1	7210	3111 B or C	D1688-90(A or B)	I-3271-85 or I-3270-85	974.27[5]
	AA furnace,	220.2 or 200.9[1g]	7211	3113 B	D1688-90(C)		
	Inductively coupled plasma[6m]	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
	Direct current plasma[6m],				D4190-82(88)		Note 36
	Colorimetric (Neocuproine), or			3500-Cu D or E			
	Bicinchoninate						Note 21
23	Cyanide - Total, ug/L:						
	Manual distillation with $MgCl_2$			4500-CN-C			
	Followed by: titrimetric,			4500-CN-D			
	Manual or	335.2	9010A	4500-CN-E	D2036-91(A)	I-3300-85	
	Automated[22] spectrophotometric, or	335.3	9010A				
	Semi-automated colorimetry	335.4[1m]	9012				
24.	Cyanide amenable to chlorination, ug/L: Manual distillation with $MgCl_2$ followed by titrimetric, manual or automated spectrophotometric	335.1	9010A	4500-CN-G	D2036-91(B)		
25.	Fluoride - Total, mg/L:						
	Manual distillation[8]			4500-F-B			
	Followed by manual or automated electrode,	340.2		4500-F-C	D1179-88(B)	I-4327-85	
	SPADNS,	340.1		4500-F-D	D1179-88(A)		

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
Ion chromatography,	300.0[1m]	9056				
Or automated complexone	340.3		4500-F-E			
26. Gold, mg/L:						
Digestion[6] followed by:						
AA direct aspiration	231.1		3111 B			
AA furnace,	231.2		3113 B			
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
27. Hardness - Total as $CaCO_3$, mg/L:						
Automated colorimetric,	130.1					
EDTA titration,	130.2		2340 C	D1126-86(92)	I-1338-85	973.52B[5]
or the sum of Ca and Mg as their respective carbonates (by ICP or						
AA direct aspiration)			2340 B			
(See Parameters 13 and 33)						
28. Hydrogen ion (pH), pH units:						
Electrometric Measurements or	150.1	9040B	4500-H+ B	D1293-84(90) (A or B)	I-1586-85	973.41[5]
Automated Electrode						Note 23
29. Iridium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	235.1		3111 B			
AA furnace, or	235.2					
Inductively coupled plasma	200.7[1g]	6010A				
30. Iron, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	236.1	7380	3111 B or C	D1068-90 (A or B)	I-3381-84	973.27[5]
AA furnace,	236.2 or 200.9[1g]	7381	3113 B	D1068-90(C)		
Inductively coupled plasma6m,	200.7[1g]	6010A	3120 B			
Direct current plasma[6m], or				D4190-82(88)		Note 36
Colorimetric (Phenanthroline)			3500-Fe D	D1068-90(D)		Note 24
31. Kjeldahl nitrogen - Total (as N), mg/L:						
Digestion and distillation	351.3		4500-N org B or C	D3590-89(A)		
Followed by titration	351.3		4500-NH_3 E	D3590-89(A)		937.46[5]
Nesslerization or	351.3		4500-NH_3 C	D3590-89(A)		
Electrode,	351.3		4500-NH_3 F or G			
Automated phenate,	351.1		4500-NH_3 H		I-4551-78[8]	
Semi-automated block digester,	351.2[1m]			D3590-89(B)		
Or potentiometric	351.4			D3590-89(A)		
32. Lead, mg/L:						
Digestion[6] followed by:						
AA direct aspiration6m,	239.1	7420	3111 B or C	D3559-90 (A or B)	I-3399-90	974.27[5]
AA furnace,	239.2 or 200.9[1g]	7421	3113 B	D3559-90(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
Direct current plasma[6m],				D4190-82(88)		Note 36
Voltametry[13] or				D3559-90(C)		
Colorimetric (Dithizone)			3500-Pb D			
33. Magnesium, mg/L:						
Digestion[6] followed by:						
Atomic absorption,	242.1	7450	3111 B	D511-92(B)	I-3447-85	974.27[5]

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

	Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
	Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
	Direct current plasma, or						Note 36
	Gravimetric			3500-Mg D			
34.	Manganese, mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration[6m],	243.1	7460	3111 B	D858-90 (A or B)	I-3454-85	974.27 5
	AA furnace,	243.2 or 200.9[1g]	7461	3113 B	D858-90(C)		
	Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
	Direct current plasma[6m],				D4190-82(88)		Note 36
	Colorimetric (Persulfate), or			3500-Mn D			920.203[5]
	Periodate						Note 25
35.	Mercury - Total[6], ug/L:						
	Cold vapor AA, manual or	245.1[1g]	7470A	3112 B	D3223-91	I-3462-85	977.22[5]
	automated, or	245.2					
35m.	Mercury - Hg(II) and organomercurials, ug/L:						
	HPLC with electrochemical detection	245.3[1g]					
36.	Molybdenum, mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration,	246.1	7480	3111 D		I-3490-85	
	AA furnace,	246.2	7481	3113 B			
	Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry, or	200.8[1g]	6020				
	Direct current plasma						Note 36
37.	Nickel, mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration[6m],	249.1	7520	3111 B or C	D1886-90 (A or B)	I-3499-85	
	AA furnace,	249.2 or 200.9[1g]		3113 B	D1886-90(C)		
	Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
	Direct current plasma[6m], or				D4190-82(88)		Note 36
	Colorimetric (Heptoxime)			3500-Ni D			
38	Nitrate (as N), mg/L:						
	Brucine sulfate, or	352.1					973.50[5], 419D[19]
	Nitrate-nitrite N minus Nitrite N (see parameters 39 and 40)						
	Ion chromatography	300.0[1m]	9056				
39	Nitrate-nitrite (as N), mg/L:						
	Cadmium reduction, manual	353.3		4500-NO_3 E	D3867-90(B)		
	or automated, or	353.2[1m]		4500-NO_3 F	D3867-90(A)	I-4545-85	
	automated hydrazine	353.1		4500-NO_3 H			
	Ion chromatography	300.0[1m]	9056				
40.	Nitrite (as N), mg/L:						
	Spectrophotometric, manual or	354.1		4500-NO_2 B			Note 27
	automated (Diazotization), or					I-4540-85	
	Ion chromatography[39]	300.0[1m]	9056				
41.	Oil and grease-Total recoverable, mg/L:						
	Gravimetric (freon extraction)	413.1	9070	5520 B			
	Gravimetric (hexane extraction)	1664					

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
42. Organic carbon - Total (TOC), mg/L:						
Combustion or oxidation,	415.1	9060	5310 B or D	D2579-85 (A or B)	973.47[5] p.142[6]	
Persulfate oxidation	415.2[1m]		5310 C			
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, automated	365.1		4500-P F		I-4601-85	973.56[5]
Or manual single reagent or	365.2		4500-P E	D515-88(A)		973.55[5]
Manual two reagent, or	365.3					
Ion chromatography	300.0[1m]	9056				
45. Osmium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	252.1	7550	3111 D			
AA furnace, or	252.2					
Inductively coupled plama	200.7[1g]	6010A				
46. Oxygen, dissolved, mg/L:						
Winkler (Azide modification)	360.2		4500-O C	D888-92(A)	I-1575-78[10]	973.45B[5]
Or electrode	360.1		4500-O G	D888-92(B)	I-1576-78[10]	
47. Palladium, mg/L:						
Digestion6 followed by:						
AA direct aspiration,	253.1		3111 B			
AA furnace,	253.2					
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
48. Phenols, ug/L:						
Manual distillation[28]	420.1		5530 B			Note 29
Followed by manual	420.1	9065	5530 D			
Or automated[22] colorimetric (4AAP), or	420.2	9066				Note 29
Semi-automated colorimetric	420.4[1m]					
49. Phosphorus (elemental), mg/L:						
Gas-Liquid chromatography						Note 30
50. Phosphorus - Total, mg/L:						
Persulfate digestion	365.2		4500-P B,5			973.55[5]
Followed by manual or	365.2 or		4500-P E			
	365.3			D515-88 (A)		
Automated ascorbic acid	365.1[1m]		4500-P F		I-4600-85	973.56[5]
Reduction, or semi-automated block digestor	365.4					
51. Platinum, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	255.1		3111 B			
AA furnace,	255.2					
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
52. Potassium, mg/L:						
Digestion[6] followed by:						
Atomic absorption,	258.1	7610	3111 B		I-3620-85	973.53[5]
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Flame photometric, or			3500-K D			
Colorimetric (cobalt nitrate)						317B[19]

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

	Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
53.	Residue - total, (total solids), mg/L:						
	Gravimetric 103-105°C	160.3		2540 B		I-3750-85	
54.	Residue - filterable, (TDS), mg/L:						
	Gravimetric, 180°C	160.1		2540 C		I-1750-85	
55.	Residue - nonfilterable, (TSS),						
	mg/L: Gravimetric,	160.2		2540 D		I-3765-85	
	103-105°C post washing of residue						
56.	Residue - settleable, mg/L:						
	Volumetric	160.5		2540 F			
	(Imhoff cone) or gravimetric						
57.	Residue - volatile mg/L:						
	Gravimetric, 550°C	160.4		2540 E[38]		I-3753-85	
58.	Rhodium, ug/L:						
	Digestion[6] followed by:						
	AA direct aspiration,	265.1		3111 B			
	AA furnace, or	265.2					
	Inductively coupled plasma	200.7[1g]	6010A				
59.	Ruthenium, ug/L:						
	Digestion[6] followed by:						
	AA direct aspiration,	267.1		3111 B			
	AA furnace, or	267.2					
	Inductively coupled plasma	200.7[1g]	6010A				
60.	Selenium, ug/L:						
	Digestion[6] followed by:						
	AA furnace,	270.2 or 200.9[1g]	7740	3113 B			
	Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
	or AA (gaseous hydride)		7741A	3114 B[37]	D3859-88(A)	I-3667-85	
61.	Silica - Dissolved, mg/L:						
	0.45 micron filtration:						
	Followed by manual or	370.1		4500-Si D	D859-88	I-1700-85	
	automated colorimetric					I-2700-85	
	(Molybdosilicate), or						
	Inductively coupled plasma[6]	200.7[1g]	6010A	3120 B			
62.	Silver[31], mg/L:						
	Digestion[6] followed by:						
	AA direct aspiration,		7760A	3111 B or C		I-3720-85	973.27[5]
	AA furnace,	200.9[1g]	7761	3113 B			
	Colorimetric (Dithizone),						319B[19]
	Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
	Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
	Or direct current plasma						Note 36
63.	Sodium, mg/L:						
	Digestion[6] followed by:						
	Atomic absorption,	273.1	7770	3111 B		I-3735-85	973.54[5]
	Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
	Direct current plasma, or						Note 36
	Flame photometric			3500-Na D	D1428-82(A)		
64.	Specific conductance, micromhos/cm						
	at 25°C: Wheatstone bridge	120.1	9050	2510 B	D1125-91(A)	I-1780-85	973.40[5]

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
65. Sulfate (as SO_4), mg/L:						
Automated colorimetric (barium chloroanilate),	375.1	9035				
Semi-automated colorimetric (methylthymol blue)	375.2[1m]	9036				
Gravimetric,	375.3		4500-SO_4^{2-} C or D			925.54[5]
Turbidimetric, or	375.4	9038		D516-90		426C[32]
Ion chromatography	300.0[1m]	9056				
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-}			
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, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	279.1	7840	3111 B			
AA furnace,	279.2 or 200.9[1g]	7841	3113 B			
Inductively coupled plasma, or	200.7[1g]	6010A				
Inductively coupled plasma-mass spectrometry	200.8[1g]	6020				
71. Tin, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	282.1	7870	3111 B		I-3850-7810	
AA furnace, or	282.2 or 200.9[1g]		3113 B			
Inductively coupled plasma	200.7[1g]	6010A				
72. Titanium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration ,	283.1		3111 D			
AA furnace,	283.2		3113 B			
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
73. Turbidity, NTU: Nephelometric	180.1[1m]		2130 B	D1889-88(A)	I-3860-85	
74. Vanadium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	286.1	7910	3111 D			
AA furnace,	286.2	7911	3113 B			
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry	200.8[1g]					
Direct current plasma, or				D4190-82(88)		Note 36
Colorimetric (Gallic acid)			3500-V D			
75. Zinc, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	289.1	7950	3111 B or C		I-3900-85	974.27[5]
AA furnace,	289.2 or 200.9[1g]	7951	3113 B			
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma-mass spectrometry,	200.8[1g]	6020				
Direct current plasma[6m],				D4190-82(88)		Note 36
Colorimetric (Dithizone), or			3500-Zn E			

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	SW-846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
Colorimetric (Zincon)			3500-Zn F			Note 36

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 (703) 487-4650.

[1s] "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91-231498, 5258 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

[1m] "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA-600/R-93-100, Environmental Protection Agency, August 1993, Office of Research and Development, Washington D.C. 20460, August 1993. Available from NTIS, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487-4650.

[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, 18th Edition, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[2m] The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

[3] "1993 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993. 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] A digestion procedure is required to solubilize suspended material and to destroy possible organic metal complexes. The required digestion procedure(s) for a particular metals analysis is listed in Table BM, Metals Digestion Procedures. Use of the graphite furnace AA technique, inductively coupled plasma, direct current plasma, as well as determination for certain elements such as arsenic, mercury, selenium, silver, and titanium require a modified digestion procedure. In all cases, the analytical method should be consulted for specific instructions and cautions.

If a digestion procedure is given in the determinative method for any of the metals in table B, and this digestion is not listed in table BM, the procedure given in the analytical method should be used however if the digestion included in one of the approved non-EPA references (e.g. "Standard Methods for the Examination of Water and Wastewater") is significantly different from one of the EPA procedures listed in table BM, than the EPA procedure from table BM should 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.

[6m] Either of the following microwave digestion procedures may be used:

"Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM corporation, P.O. Box 200, Mattews, North Carolina 28106-0200, April 16, 1992. Available form the CEM Corporation.

"Test Methods for Evaluating Solid Waste", SW-846 method 3015. United States EPA SW-846, 3rd Edition. Footnote 11 lists the complete reference.

[7] SW-846 series 6000 and 7000 methods include SW-846 method 7000A, the general AA method description.

[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] "Test Methods for Evaluating Solid Waste", 3rd Edition, SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including July 1992, August 1993, September 1994 and January 1995 updates, Washington D.C. 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington DC, (202) 512-1800.

[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 Na2S2O3 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, Fison Instruments, Inc., 32 32 Commerce Center, Cherry Hill Drive, Danvers MA 01923.

[37] Use the digestion given in the method.

[38] The temperature must be maintained between 500-550 °C, and not the temperature listed in the method.

[39] Nitrate-nitrite determinations by ion chromatography must be analyzed within 48 hours.

SECTION 30. NR 219.04 Table BM is created to read:

TABLE BM

METALS DIGESTION PROCEDURES

Analysis	SW-846[1]	EPA[2]	EPA[3]
Dissolved Metals[4]	3005A, 3040A[10]	----------	4.1.1
Suspended Metals[5]	3005A	----------	4.1.2
Total Metals[6]	3010A, 3020A[11], 3050A[10], 3051A[10]	----------	4.1.3
Total Recoverable Metals[7]	3005A	200.2	4.1.4
Acid Soluble Metals[8]	----------	200.1[12]	----------
Availible Metals[9]	3015[13]	----------	----------

TABLE BM NOTES

[1] "Test Methods for Evaluating Solid Waste", 3rd Edition, SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington D.C. 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington DC 20402, (202) 512-1800.

[2] "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91-231498, 5258 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

[3] "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 (703) 487-4650.

[4]"Dissolved metals" means those constituents of a sample that will pass through a 0.45 micron membrane filter prior to sample acidification.

[5]"Suspended metals" means the concentration of metals determined in the portion of a sample retained by a 0.45 micron membrane filter prior to acidification.

[6]"Total metals" means the concentration of metals determined on a solid sample or unfiltered aqueous sample following a vigorous digestion, or alternatively the sum of the the metals determined in both the dissolved and suspended fractions.

[7]"Total recoverable metals" means the concentration of metals determined on an unfiltered sample following treatment with hot dilute mineral acid.

[8]"Acid soluble metals" means those constituents of a sample that will pass through a 0.45 micron membrane filter after the sample has been adjusted to pH 1.75 and held for 16 hours. This method is applicable to arsenic, cadmium, chromium, copper, and lead.

[9]"Availible metals" are equivalent to "total metals". SW-846 lists method 3015 as a preparation for available metals.

[10]These methods are for total metals analysis of sediment, sludge, and soil samples and do not apply to wastewater. The required analytical methodology for metals in wastewater sludge is given in Table EM.

[11]Method 3020 is applicable for analysis by GFAA. Method 3010 requires sample acidification with HCl.

[12]Method 200.1 is only applicable for As, Cd, Cr, Cu and Pb.

[13]This method is a microwave-assisted acid leachate digestion.

SECTION 31. NR 219.04 Table C is repealed and recreated to read:

TABLE C

List of Approved Test Procedures for Non-Pesticide Organic Compounds in Wastewater

Parameter	EPA Method Number[1,6] GC	EPA Method Number[1,6] GC/MS	Standard Methods[8,13]	SW-846 Method Number[11,12] GC capillary	SW-846 Method Number[11,12] GC pkd[14]	SW-846 Method Number[11,12] GC/MS capillary	SW-846 Method Number[11,12] GC/MS pkd[14]	Other
I. VOLATILES		**624[3]**		**8021A**	**——**	**8260A**	**8240B**	
A. Halogenated volatiles	**601**	**1624**	**6230 B, 6210 B**	**——**	8010B	**——**	**——**	
Bromodichloromethane								
Bromoform								
Bromomethane								
Carbon tetrachloride								Note 2, p.130
Chloroethane								
Chloroform								Note 2, p.130
Chloromethane								
Dibromochloromethane								
Dichlorodifluoromethane		——	not 6210 B					
1,1-Dichloroethane								
1,2-Dichloroethane								
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
1,2-Dichloropropane								
cis-1,3 Dichloropropene								
trans-1,3-Dichloropropene								
Methylene chloride								Note 2, p.130
1,1,2,2-Tetrachloroethane								Note 2, p.130
Tetrachloroethene								Note 2, p.130
1,1,1-Trichloroethane								
1,1,2-Trichloroethane								Note 2, p.130
Trichloroethene								
Trichlorofluoromethane		——						
Vinyl chloride								
B. Aromatic volatiles	**602**		**6220 B**	**——**	**8020A**	**——**	**——**	
Benzene		1624	6210 B					
Chlorobenzene	601	1624	6210 B, 6230 B					Note 2, p.130
1,2-Dichlorobenzene	601, 612	625, 1625	6230 B, 6410 B					
1,3-Dichlorobenzene	601, 612	625, 1625	6230 B, 6410 B					
1,4-Dichlorobenzene	601, 612	625, 1625	6230 B, 6410 B					
Ethylbenzene		1624	6210 B					
Toluene		1624	6210 B					
C. Other volatiles	**603**	**1624,624[3]**		**8030A**	**——**	**8260A**	**8240B**	
Acrolein								LC: 8315 (SW-846)
Acrylonitrile				8031				LC: 8316 (SW-846)
II. PHENOLS	**604**	**625, 1625**	**6410 B, 6420 B**	**——**	**8040A**	**8270B**	**8250A**	
4-Chloro-3-methylphenol								
2-Chlorophenol								
2,4-Dichlorophenol								
2,4-Dimethlyphenol								
2,4-Dinitrophenol								
2-Methyl-4,6-dinitrophenol								
2-Nitrophenol								
4-Nitrophenol								
Pentachlorophenol								Note 2, p.140
Phenol								
2,4,6-Trichlorophenol								
III. PHTHALATE ESTERS	**606**	**625, 1625**	**6410 B**	**8061**	**8060**	**8270B**	**8250A**	
Benzyl butyl phthalate								
Bis(2-ethylhexyl)phthalate								

Diethyl phthalate								
Dimethyl phthalate								
Di-n-butyl phthalate								
Di-n-octyl phthalate								
IV. NITROSAMINES	**607**	**625, 1625**	**6410 B**	**----**	**8070**	**8270B**	**8250A**	
N-Nitrosodimethylamine		note 4						
N-Nitrosodi-n-propylamine								
N-Nitrosodiphenylamine		note 4						
V. POLYCHLORINATED BIPHENYLS	**608**	**625**	**6410 B**	**8081**	**8080A**	**8270B**	**8250A**	**Note 2, p.43**
PCB-1016								
PCB-1221								
PCB-1232								
PCB-1242								
PCB-1248								----
PCB-1254								
PCB-1260								
VI. NITROAROMATICS &CYCLIC KETONES	**609**	**625, 1625**	**6410 B**	**----**	**8090**	**8270B**	**8250A**	
2,4-Dinitrotoluene								
2,6-Dinitrotoluene								
Isophorone								
Nitrobenzene								
VII. POLYNUCLEAR AROMATIC HYDROCARBONS	**610/FID**	**625, 1625**	**6410 B, 6440 B**	**----**	**8100**	**8270B**	**8250A**	**Note 9; 610, LC: 8310 (SW-846)**
Acenaphthene								
Acenaphthylene								
Anthracene								
Benzo(a)anthracene								
Benzo(a)pyrene								
Benzo(b)fluoranthene								
Benzo(g,h,i)perylene								
Benzo(k)fluoranthene								
Chrysene								
Dibenzo(a,h)anthracene								
Fluoranthene								
Fluorene								
Ideno (1,2-3-cd)pyrene								
Naphthalene				8021A				
Phenanthrene								
Pyrene								
VIII. HALOETHERS	**611**	**625, 1625**	**6410 B**	**----**	**8110**	**8270B**	**8250A**	
Bis(2-chloroethoxy) methane								
Bis(2-chloroethyl)ether								
4-Bromophenylphenyl ether								
4-Chlorophenylphenyl ether								
2,2-Oxybis (1-chloropropane)								
IX. CHLORINATED HYDROCARBONS	**612**	**625, 1625**	**6410 B**	**8121**	**8120A**	**8270B 8260A**	**8250A, 8240A**	
Benzyl chloride	----	----	----		8010B	not 8270B	not 8250A	Note 2, p.130; Note 5, p.S102
2-Chloronaphthalene						not 8260A	not 8240A	8410 (SW-846)
Epichlorohydrin	----	----	----		8010B	not 8270B	not 8250A	Note 2, p.130; Note 5, p.S102
Hexachlorobenzene				8081		not 8260A	not 8240A	8410 (SW-846)
Hexachlorobutadiene				8021A			not 8240A	8410 (SW-846)
Hexachlorocyclopentadiene		note 4		8081		not 8260A	not 8240A	8410 (SW-846)
1,2,4-Trichlorobenzene				8021A			not 8240A	Note 2, p.130
Hexachloroethane							not 8240A	8410 (SW-846)

Benzidine	note 4		----	----	not 8260A	not 8240A	LC: 605
3,3-Dichlorobenzidine			----	----	not 8260A	not 8240A	
X. POLYCHLORINATED DIBENZO-P-DIOXINS AND FURANS	**1613 A[7]**		----	----	**8280, 8290**	----	
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin							
1,2,3,4,6,7,8-Heptachlorodibenzofuran							
1,2,3,4,7,8,9-Heptachlorodibenzofuran							
1,2,3,4,7,8-Hexachlorodibenzo-p -dioxin							
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin							
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin							
1,2,3,4,7,8-Hexachlorodibenzofuran							
1,2,3,6,7,8-Hexachlorodibenzofuran							
1,2,3,7,8,9-Hexachlorodibenzofuran							
2,3,4,6,7,8-Hexachlorodibenzofuran							
Octachlorodibenzo-p-dioxin							
Octachlorodibenzofuran							
1,2,3,7,8-Pentachlorodibenzo-p-dioxin							
1,2,3,7,8-Pentachlorodibenzofuran							
2,3,4,7,8-Tetrachlorodibenzo-p-dioxin							
2,3,7,8-Tetrachlorodibenzo-p-dioxin	613[5m]						Note 10
2,3,7,8-Tetrachlorodibenzofuran							

TABLE C NOTES

[1] 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

[2] "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.

[3] 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.

[4] 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.

[5] "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.

[5m] 625 Screening only.

[6] 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.

[7] 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.

[8] "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, 18th Edition, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[9] Method D4657-92, "Annual Book of Standards- Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[10] Method D4675-92, "Annual Book of Standards- Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[11] "Test Methods for Evaluating Solid Waste", 3rd Edition. SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512-1800.

[12] SW-846 methods 8021, 8061, 8081, and 8121 require one of the following sample preparation (extraction/clean-up) procedures: 3500/3510 (liquid-liquid extraction), 3500/3520 (continuous liquid-liquid extraction), or 5030 (purge and trap method). The required sample preparation procedure is given in the determinative procedure. Method 8021 requires 5030 (purge and trap). Methods 8081 and 8121 require either 3500/3510 or 3500/3520 in addition to 3600. Method 8061 requires 3510. For methods 8021, 8061, 8081, and 8121 see also SW-846 method 8000A.

[13] The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

[14] In order to reference these methods, the laboratoy must use a packed column for the GC separations.

SECTION 32. NR 219.04 Table D is repealed and recreated to read:

TABLE D

List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter	Method	EPA[2,7]	SW-846[4,8] pkd[11]	SW-846[4,8] cap.	Standard Methods[B,9]	ASTM[C]	Other
1. Aldrin	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS	625	8250A	8270B	6410 B		
2. Ametryn	GC						Note 3, p.83; Note 6, p.S68
3. Aminocarb	HPLC						Note 10
4. Atraton	GC						Note 3, p.83; Note 6, p.S68
5. Atrazine	GC		8140	8141A			Note 3. p.83; Note 6, p.S68
6. Azinphos methyl	GC		8140	8141A			Note 3. p.25; Note 6, p.S51
	GC/MS		8250A	8270B			
7. Barban	HPLC						Note 10
	GC/MS		8250A	8270B			
8. α-BHC	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7
	GC/MS	625[5]	8250A	8270B	6410 B		
9. β-BHC	GC	608	8080A	8081	6630 C	D3086-90	
	GC/MS	625	8250A	8270B	6410 B		
10. δ-BHC	GC	608	8080A	8081	6630 C	D3086-90	
	GC/MS	625[5]	8250A	8270B	6410 B		
11. γ-BHC (Lindane)	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS	625	8250A	8270B	6410 B		
12. Captan	GC				6630 B	D3086-90	Note 3. p.7.
	GC/MS		8250A	8270B			
13. Carbaryl	HPLC						Note 10
	GC/MS		8250A	8270B			
14. Carbophenothion	GC		8140	8141A			Note 4, p.30; Note 6, p.S73.
	GC/MS		8250A	8270B			
15. Chlordane	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7
	GC/MS	625	8250A	8270B	6410 B		
16. Chloropropham	HPLC						Note 10
17. 2,4-D	GC		8150B	8151	6640 B		Note 3, p.115; Note 4, p.35.
18. 4,4'-DDD	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3. p.7; Note 4, p.30.
	GC/MS	625	8250A	8270B	6410 B		
19. 4,4'-DDE	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30.
	GC/MS	625	8250A	8270B	6410 B		
20. 4,4'-DDT	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS	625	8250A	8270B	6410 B		
21. Demeton-O	GC		8140	8141A			Note 3, p.25; Note 6, p.S51.
	GC/MS		8250A	8270B			
22. Demeton-S	GC		8140	8141A			Note 3, p.25; Note 6, p.S51.
	GC/MS		8250A	8270B			
23. Diazinon	GC		8140	8141			Note 3, p.25; Note 4, p.30;Note 6, p.S51
24. Dicamba	GC		8150B	8151			Note 3, p.115
25. Dichlofenthion	GC		8140	8141			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	8080A	8081	6630 B & C		Note 3, p.7; Note 4, p.30
	GC/MS	625	8250A	8270B	6410 B		
29. Dioxathion	GC		8140	8141A			Note 4, p.30; Note 6, p.S73
	GC/MS		8250A	8270B			
30. Disulfoton	GC		8140	8141A			Note 3, p.25; Note 6, p.S51
	GC/MS		8250A	8270B			
31. Diuron	HPLC						Note 10
32. Endosulfan I	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7
	GC/MS	625[5]	8250A	8270B	6410 B		
33. Endosulfan II	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7
	GC/MS	625[5]	8250A	8270B	6410 B		

TABLE D

List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter	Method	EPA[2,7]	SW-846[A,8] pkd[11]	SW-846[A,8] cap.	Standard Methods[B,9]	ASTM[C]	Other
34. Endosulfan sulfate	GC	608	8080A	8081	6630 C		
	GC/MS	625	8250A	8270B	6410 B		
35. Endrin	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS	625[5]	8250A	8270B	6410 B		
36. Endrin aldehyde	GC	608	8080A	8081		D3086-90	
	GC/MS	625	8250A	8270B	6410 B		
37. Ethion	GC		8140	8141A			Note 4, p.30; Note 6, p.S73
	GC/MS		8250A	8270B			
38. Fenuron	HPLC						Note 3, p.104; Note 6, p.S64
39. Fenuron-TCA	HPLC						Note 10
40. Heptachlor	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS	625	8250A	8270B	6410 B		
41. Heptachlor epoxide	GC	608	8080A	8081	6630 B	D3086-90	Note 3, p.7; Note 4, p.30; Note 6 p.S73
	GC/MS	625	8250A	8270B	6410 B		
42. Isodrin	GC		8080A	8081			Note 4, p.30; Note 6, p.S73
	GC/MS		8250A	8270B			
43. Linuron	HPLC						Note 10
44. Malathion	GC		8140	8141A	6630 C		Note 3, p.25; Note 4, p.30; Note 6, p.S51
	GC/MS		8250A	8270B			
45. Methiocarb	HPLC						Note10
46. Methoxychlor	GC		8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS		8250A	8270B			
47. Mexacarbate	HPLC						Note 10
	GC/MS		8250A	8270B			
48. Mirex	GC		8080A	8081	6630 B & C		Note 3, p.7
	GC/MS		8250A	8270B			
49. Monuron	HPLC						Note 10
50. Monuron-TCA	HPLC						Note 10
51. Neburon	HPLC						Note 10
52. Parathion methyl	GC		8140	8141A	6630 C		Note 3, p.25; Note 4, p.30
	GC/MS		8250A	8270B			
53. Parathion ethyl	GC		8140	8141A	6630 C	D3086-90	Note 3, p.25
	GC/MS		8250A	8270B			
54. PCNB	GC		8080A	8081	6630 B & C		Note 3, p.7
	GC/MS		8250A	8270 B			
55. Perthane	GC		8080A	8081		D3086-90	
56. Prometon	GC						Note 3, p.83; Note 6, p.S68
57. Prometryn	GC						Note 3, p.83; Note 6, p.S68
57. Propazine	GC						Note 3, p.83; Note 6, p.S68
58. Propham	HPLC						Note 10
59. Propoxur	HPLC						Note 10
60. Secbumeton	HPLC						Note 10
61. Siduron	HPLC						Note 10
62. Simazine	GC		8140	8141A			Note 3, p.83; Note 6, p.S68
63. Strobane	GC		8080A	8081	6630 B & C		Note 3, p.7
64. Swep	HPLC						Note 10
65. 2,4,5-T	GC		8150B	8151	6640 B		Note 3, p.115; Note 4, p.35
66. 2,4,5-TP (Silvex)	GC		8150B	8151	6640 B		Note 3, p.115
67. Terbuthylazine	GC						Note 3, p.83; Note 6, p.S68
68. Toxaphene	GC	608	8080A	8081	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
	GC/MS	625	8250A	8270B	6410 B		
70. Trifluralin	GC		8080A	8081	6630 B		Note 3, p.7
	GC/MS		8080A	8270B			

TABLE D NOTES

[A] "Test Methods for Evaluating Solid Waste", 3rd Edition. SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washingtion, DC 20402, (202) 512-1800.

[B] "Standard Methods for the Examination of Water and Wastewater", 18th Edition, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[C] "Annual Book of Standards- Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from the 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 D, where entries are listed by chemical name and type.

[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.

[8] Some of these methods require a preliminary extraction. Methods 8141 A and 8081 require the use of either SW-846 method 3500/3510 or 3500/3520. Methods 8151 and 8270 B include the extraction steps necessary for most compounds. For methods 8081, 8141, and 8151 see also SW-846 method 8000 A and 3600.

[9] The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

[10] HPLC method 623 from "Methods for Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater", EPA 440/1-83/079-C, United States Environmental Protection Agency. Available from National Technical Information Service, 5258 Port Royal Road, Springfield, Virginia, 22161 (703) 487-4650.

[11] In order to reference these methods, the laboratoy must use a packed column for the GC separations.

SECTION 33. NR 219.04 Table E, (note 2) and (note 3) are amended to read:

TABLE E
List of Approved Radiological Test Procedures in Wastewater

	Parameter and Units	Method	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]
1.	Alph-Total, pCi per liter	Proportional or Scintillation Counter	900.0	~~703~~ 7110 B	D1943-~~81~~90	pp. 75 and 78[5]
2.	Alpha-Counting error, pCi per liter	Proportional or Scintillation Counter	Appendix B	~~703~~ 7110 B	D1943-~~81~~90	p. 79
3.	Beta-Total, pCi per liter	Proportional Counter	900.0	~~703~~ 7110 B	~~D1943-81~~ D1890-90	pp. 75 and 78[5]

4.	Beta-Counting error, pCi	Proportional Counter	Appendix B	~~703~~ 7110 B	~~D1943-81~~ D1890-90	p. 79
5.	(a) Radium-Total	Proportional Counter	903.0	~~705~~ 7500Ra B	D2460-~~70~~90	
	(b) ^{226}Ra, pCi per liter	Scintillation Counter	903.1	~~706~~ 7500Ra C	D3454-~~79~~91	p. 81

NR 219.04, Table E note [2]"Standard Methods for the Examination of Water and Wastewater", 17th or 18th Edition, Joint Editorial Board, American Public Health 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.

NR 219.04, Table E note [3]"~~1991~~1993 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, ~~1980~~1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

SECTION 34. NR 219.04 Table EM (header) and (note 4) are amended to read:

TABLE EM

Approved Analytical Methods for Sludge

Parameter	Digestion[11]	Method	Method Number

NR 219.04 Table EM note [4]"~~1991~~ Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993, 1916 Race Street, Philadelphia, PA 19103. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

SECTION 35. NR 219.04 Table EM (note 9) is created to read:

NR 219.04 Table EM note [9] If an alternative digestion procedure is specified in the analytical method, the digestion in the method shall be used. In all cases, consult the analytical method for special requirements and cautions. SW-846 method 3051 is an acceptable alternate digestion procedure to SW-846 method 3050A.

SECTION 36. NR 219.04 Table F is repealed and recreated to read:

TABLE F

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR WASTEWATER

Parameter Number	Parameter Name	Container[1]	Preservation[2,3]	Maximum holding time[4]
TABLE A - Bacterial Tests:				
1-5.	Bacteria	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$[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
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	G	Cool, 4°C, HCl or H_2SO_4 or H_3PO_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
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, or Quartz	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 NaOH 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 B - Metals[7]:				
10.	Boron	P, or Quartz	HNO_3 to pH<2	6 months

18	Chromium VI	P,G	Cool, 4°C	24 hours
35 & 35m	Mercury	P,G, or Teflon	HNO_3 to pH<2	28 days
71	Tin	P	HCl or HNO_3 to pH<2	6 months
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 Cr VI, Sn, Hg, & B)	P,G	HNO_3 to pH<2	6 months

TABLE C - Organic Tests[8]:

IA	Purgeable halocarbons	G, Teflon-lined septum	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5]	14 days
IB	Purgeable aromatics	G, Teflon-lined septum	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5], HCl to pH<2	14 days
IC	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
II	Phenols[11]	G, Teflon-lined cap	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
IX	Benzidines (Benzidine and 3,3- Dichlorobenzidine)[11]	G, Teflon-lined cap	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5]	7 days after extraction[13]
III	Phthlate esters[11]	G, Teflon-lined cap	Cool, 4 °C	7 days until extraction; 40 days after extraction
IV	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
V	PCBs[11]	G, Teflon-lined cap	Cool, 4 °C	7 days until extraction; 40 days after extraction
VI	Nitroaromatics, cyclic ketones and isophorone[11]	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
VII	Polynuclear aromatic hydrocarbons[11]	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
VIII	Haloethers[11]	G, Teflon-lined cap	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
IX	Chlorinated hydrocarbons[11]	G, Teflon-lined cap	Cool, 4 °C	7 days until extraction; 40 days after extraction
X	Chorinated Dioxans 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 E - 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 F - 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] All samples requiring preservation at 4 °C must be cooled immediately after collection, and the temperature of the samples shall be documented upon receipt at the laboratory. If the samples are shipped in crushed or cube ice (not "blue ice" packs) and solid ice is still present in the cooler, the lab may simply report the samples as "received on ice". If the ice has melted, the lab must report the either the temperature of the meltwater or of a temperature blank. A temperature blank is defined as an aliquot of deionized water, in an appropriate sample container, which is transported along with the samples. If sampling teams use "blue ice" packs, it is necessary to pre-chill all sample containers to at least 4 degrees celsius with ice or refrigeration prior to shipping. Since shipping simply with "blue ice" packs does not insure that samples are maintained at the appropriate temperatures, the sample collector must submit a temperature blank when using these ice packs for shipping. 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 mail, 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 (HNO3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H2SO4) 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. For other composite samples, the holding time commences immediately after the samples are removed from the composite sampler. The time the sample spends in the sampler during collection does not count towards the maximum holding time. 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 219.05). 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 37. NR 219.05 and NR 219.06 are amended 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.~~The department may approve the use of an alternate test procedure on a case-by-case basis if the criteria for approval of the alternate procedure established in s. NR 149.12 are met. If the department or the EPA approves an alternate test procedure, it shall be considered equivalent to the approved method.

NR 219.06 LABORATORY CERTIFICATION OR REGISTRATION. (intro.) 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~~ a WPDES permit shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

(1) Temperature,

(2) Turbidity,

(3) Bacteria tests in wastewater effluent and sludges,

(4) pH,

(5) Chlorine residual,

(6) Specific conductance,

(7) Physical properites of soils and sludges,

(8) Nutrient test of soils and sludges,

(9) Flow measurements.

SECTION 38. NR 700.13 and (note) are created to read:

NR 700.13 SAMPLE PRESERVATION AND ANALYSIS. (1) PETROLEUM PRODUCTS. Soil or groundwater analyses for gasoline range organics or diesel range organics conducted for compliance with chs. NR 700 to 736 shall be completed in accordance with the "Modified GRO, Method for Determining Gasoline Range Organics" and the "Modified DRO, Method for Determining Diesel Range Organics", as specified in s. NR 149.03(5).

(2) VOLATILE ORGANIC COMPOUNDS. (a) Soil samples collected for analysis of volatile organic compounds for compliance with chs. NR 700 to 736 shall be preserved in methanol immediately after collection unless the samples are stored in a device which insures sample integrity. Samples stored in a suitable device, including brass tubes and EnCore™ samplers, shall be preserved in methanol according to sub. (3). The department may approve alternate storage devices on a case-by-case basis prior to use in the field. Samples shall be preserved and handled as specified in section 8 and Table 1 of the "Modified GRO, Method for Determining Gasoline Range Organics".

(b) Methanol-preserved soil samples shall be extracted in the vial by shaking for 2 minutes and sonicating in an ultrasonic water bath for 20 minutes. After sonication, an aliquot not larger than 100 microliters shall be removed and introduced into a purge and trap system as in par. 7.3.3.2.4 of method 5030A in "Test Methods for Evaluating Solid Waste (SW-846)".

(c) Methanol-preserved soil samples shall be analyzed by gas chromatography or gas chromatography/mass spectrometry using capillary columns. Suitable analytical methods are found in SW-846.

(3) HOLDING TIMES. Maximum holding times for soils shall be in accordance with the following table:

Analysis Method	Sample Storage	Maximum Holding Times from Date and Time of Collection			
		Solvent Addition	Shipping	Extraction	Analysis
GRO/VOC/PVOC	VOC vial	immediately	4 days	21 days	21 days
	Brass Tube	within 2 hours	4 days	21 days	21 days
	EnCore™	within 48 hours	40 hours	21 days	21 days
VOC/PVOC Confirmation	NA	NA	NA	NA	28 days
DRO	VOC vial	72 hours	72 hours	47 days	47 days
	EnCore™	72 hours	72 hours	47 days	47 days

Note: The "Modified GRO, Method for Determining Gasoline Range Organics" (WI-PUBL-SW-141) and "Modified DRO, Method for Determining Diesel Range Organics" (WI-PUBL-SW-140) are available from the Department of Natural Resources, Emergency and Remedial Response Section, 101 S. Webster St., Madison, WI 53707.

The foregoing rules were approved and adopted by the State of Wisconsin Natural Resources Board on September 28, 1995.

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., except for sub. NR 149.15(3) which shall take effect on January 1, 1997.

Dated at Madison, Wisconsin November 22, 1995.

STATE OF WISCONSIN
DEPARTMENT OF NATURAL RESOURCES

By George E. Meyer
George E. Meyer, Secretary

(SEAL)

WISCONSIN
DEPT. OF NATURAL RESOURCES

George E. Meyer
Secretary

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

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November 21, 1995

Mr. Gary L. Poulson
Assistant Revisor of Statutes
131 West Wilson Street - Suite 800
Madison, WI

Dear Mr. Poulson: Gary

Enclosed are two copies, including one certified copy, of State of Wisconsin Natural Resources Board Order No. TS-22-95. These rules were reviewed by the Assembly Committee on Natural Resources and the Senate Committee on Environment and Energy pursuant to s. 227.19, Stats. Summaries of the final regulatory flexibility analysis and comments of the legislative review committees are also enclosed.

You will note that this order takes effect following publication. Kindly publish it in the Administrative Code accordingly.

Sincerely,

George

George E. Meyer
Secretary

Enc.