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(FORM UPDATED: 08/11/2010)

WISCONSIN STATE LEGISLATURE ... PUBLIC HEARING - COMMITTEE RECORDS

2009-10

(session year)

Joint

(Assembly, Senate or Joint)

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* Contents organized for archiving by: Stefanie Rose (LRB) (June 2012)

NOTICE TO PRESIDING OFFICERS OF PROPOSED RULEMAKING

Pursuant to s. 227.19, Stats., notice is hereby given that final draft rules are being submitted to the presiding officer of each house of the legislature. The rules being submitted are:

Board Order Number: SS-16-08

Clearinghouse Number: 08-076

Subject of Rules: Approved Analytical Methods

Date of Transmittal: January 22, 2009

Send a copy of any correspondence or notices pertaining to the rule to:
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An electronic copy of the proposed rule submittal may be obtained by contacting Diane Drinkman, Audit Chemist, at (608) 264-8950 or diane.drinkman@wisconsin.gov.

REPORT TO LEGISLATURE

NR 219, Wis. Adm. Code

Board Order No. SS-16-08
Clearinghouse Rule No. 08-076

Basis and Purpose of the Proposed Rule

On March 12, 2007, EPA published "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule". In addition, on March 26th, 2007, "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for Biological Pollutants in Wastewater and Sewage Sludge; Final Rule" was published. In these final rules, EPA updated the approved analytical test methods to be used in the National Pollutant Discharge Elimination System (NPDES) program, which regulates discharges from industrial and municipal wastewater treatment facilities. Wisconsin must incorporate these changes to maintain delegated authority for the Clean Water Act.

Summary of the rule

The following provisions, changes and requirements are implanted through the proposed rules:

- 1) Sample Preservation Procedures:** The required temperature for sample preservation was updated throughout the chapter to less than or equal to 6°C, to be consistent with the updated federal requirements. The rule also clarified maximum holding times, addressed potential interferences, and extended holding times for specific analytes. Table F, Required Containers, Preservation Techniques, and Holding Time for Wastewater was repealed and recreated to incorporate additional modifications in federal rule.
- 2) Analytical Methods Tables:** The rule retained approximately 500 methods from the previous version, deleted 62 and incorporated 367 additional analytical methods in the following tables: Table A, List of Approved Biological Analytical Methods, Table B, List of Approved Inorganic Analytical Methods in Wastewater, Table C, List of Approved Analytical Methods for Non-Pesticide Organic Compounds, Table D, List of Approved Analytical Methods for Pesticides in Wastewater, Table E, List of Approved Radiological Analytical Methods for Wastewater and Table EM, List of Approved Analytical Methods for Sludge. The rule also removed analytical methods that utilized mercury-containing reagents.

The incorporated methods included those approved through the Office of Water's Alternative Test Procedure program, updated references from "2007 Annual Book of Standards" by ASTM, on-line and the 21st Edition of "standard Methods for the Examination of Water and Wastewater", "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Updates III and IV", and several others developed by instrument manufacturers.

Summary of Public Comments

Hearing comments were categorized into three areas of concern: clarification and correction of approved analytical methods, sample preservation and holding time, and other comments.

Clarification and Correction of Approved Analytical Methods

- **COMMENT:** Requested clarification regarding the use of the luminescence technique for Biochemical Oxygen Demand (BOD₅) and Carbonaceous Biochemical Oxygen Demand (CBOD₅) determinations.
RESPONSE: Approved BOD methods direct laboratories to use methods for dissolved oxygen, to determine oxygen depletion. The approved methods for dissolved oxygen, parameter 47 in table B, includes three analytical technologies- Winkler (azide modification), electrode or luminescence. Laboratories may use any of these methods for the determination of dissolved oxygen during BOD and Carbonaceous BOD analyses.
- **COMMENT:** For Table B, parameter 21- color, why is 2120 E the only cited method versus 2120B or 2120D when comparing the 18-21st editions and on-line version of *Standard Methods for the Analysis of Water and Wastewater*.
RESPONSE: For color analyses, EPA only identified *Standard Methods* 2120 B-01 as equivalent to approved methods in the Alternate Test Procedure approval. The on-line versions of methods 2120 C and 2120 E differ dramatically from those previously published in *Standard Methods*. Although EPA only approved a single on-line version, Table B includes thirteen additional approved methods for this parameter.
- **COMMENT:** The prior version of NR 219 included a separate table for metals digestions. The proposed revision drops that table and uses footnotes to address digestion. We find it confusing to determine whether our current practice for silver digestion is allowable under the draft NR 219. We ask that the language be changed to clarify and allow our current practice.
RESPONSE: Metals digestion procedures are contained in Table EM of ch. NR 219. The department did not delete or modify the content of this table in this update effort. Table EM will be included, in the updated version of this chapter, unchanged, when rulemaking is completed. The intent of the footnotes in table B is that laboratories must use an alternative digestion if they anticipate silver concentrations in excess of 1 mg/L.
- **COMMENT:** On Page 14 – Parameter 25 – Fluoride. The ISE method is “C” not “B”. B is the distillation step.
RESPONSE: The table has been corrected to read “4500-F C [18th, 19th, 20th, 21st]” under the heading “Standard Methods”.
- **COMMENT:** On Page 15 – Parameter 28 – pH. Standard method Online is missing a zero. It should read 4500- H⁺ B-00.
RESPONSE: The table has been corrected to read “4500- H⁺ B-00 under the heading “Standard Methods Online”.
- **COMMENT:** On Page 19 – Parameter 40 - Standard method Online is missing a zero. It should read 4500-NO₂ B-00.
RESPONSE: The table has been corrected to read “4500- NO₂ B-00 under the heading “Standard Methods Online”.
- **COMMENT:** Table EM appears to list ICP method citations under both EPA and Standard Methods columns for Gaseous Hydride.
RESPONSE: The content of table EM has been corrected to indicate the only approved method for Selenium by Gaseous Hydride is Method 7471A, from “Test Methods for Evaluating Solid Water, Physical/Chemical Methods,” SW-846, September 1994.

Sample Preservation and Holding Times

- **COMMENT:** Composite samples are required to be maintained at <6°C, and sample temperature must be documented upon receipt by the laboratory. Although the intent of these statements is to ensure sample integrity during transport, we believe this requirement is unnecessary when samples

are submitted to the laboratory within 15 minutes of collection and request the note be removed or modified. Our industrial facility's final effluent stream has a temperature of 30-35°C during the warmer months of the year and no amount of refrigeration will bring these samples to the required temperature within 15 minutes of collection. We believe that documenting that the composite samples and/or refrigerators are maintained at temperatures <4°C should be sufficient.

RESPONSE: It would likely take several hours for your facility's composite sample to drop 25°C (~50°F) or more to literally meet these temperature requirements. Provided that your composite samples are collected with a refrigerated autosampler and then either analyzed immediately or stored at ≤6°C upon arrival at the laboratory, they would be considered "properly preserved" under these requirements.

- **COMMENT:** Please provide guidance on priority when there are discrepancies between NR 149 and NR 219, such as the determination of sample temperature upon receipt, and to consider this when additional revision are made.

RESPONSE: Sample preservation procedures, specified as ss. NR 219.04(2) Wis Adm. Code require immediate cooling to ≤6°C after sample collection and that temperature be maintained during shipping. s. NR 219.04(3) Wis Adm. Code, requires that samples not cooled during collection be chilled to ≤6°C prior to shipping with a temperature blank.

The sample preservation and holding time requirements listed in s. NR 149.46(4)a, Wis. Adm. Code, establishes that sample preservation and hold time requirements identified in state or federal regulations take precedence over that contained in analytical methods or authoritative sources. The following subsection, b., specifies that samples are properly preserved if, they are either surrounded by ice or actual temperature of a sample, temperature blank or melt water in the shipping container is between 0-6°C. This language mirrors that contained in ch. NR 219, Wisc. Adm. Code. In addition, permittees may petition the EPA Region V Administrator for a variance from prescribed sample preservation procedures applicable to samples from a specific discharge. The WDNR Laboratory Certification Program may consider clarification of this issue in future revisions of ch. NR 149, Wis Adm. Code.

- **COMMENT:** The language contained in Footnote 6, Table F, which addresses required preservation for cyanide determinations is complex and confusing, especially when dealing with unknown samples. Please provide clarification and guidance on the proper procedures for preserving samples for cyanide.

RESPONSE: The language contained in footnotes 5 and 6, were expanded by EPA in the final federal rule, which was based on information gathered during the development of new cyanide methods approved in this rulemaking, and information collected from various commenters and experts in cyanide analyses. All samples for WPDES compliance must be screened for the presence of sulfide at the time of sample collection. Laboratories that perform WPDES-required cyanide analyses must either analyze samples that have been preserved to pH>12 and ≤6°C within 48 hours or document that the presence and removal of all interferences have been carried out for those samples preserved to pH>12 and ≤6°C and analyzed within 14 days. When reporting cyanide results, laboratories should identify any interferences that were removed after sampling or during analytical processes.

The department agrees issuance of guidance that explains interference screening and mitigation processes will develop such materials to assist the regulated community.

- **COMMENT:** I would suggest that all laboratories have a specific data qualifier for exceeding hold times of 15 minutes, or at a minimum have a disclaimer regarding holding times for specific laboratory tests or sample preparation steps.

RESPONSE: When field testing is performed after the hold time has been exceeded, these measurements cannot be used to demonstrate compliance with a permit. The only exception would be if a facility's permit specifies "Lab pH", which assumes that the data was not generated within 15 minutes of collection.

Samples that are not immediately analyzed must also be properly preserved. This includes addition of chemical preservatives or storage at $\leq 6^{\circ}\text{C}$. Many commercial laboratories provide pre-preserved sample containers to ensure proper preservation after sample is added. Samples that are shipped to external laboratories must also contain sufficient volume of ice to ensure sample temperatures do not exceed 6°C on arrival. When reporting results of samples that have been improperly preserved, data must be qualified.

The WDNR Laboratory Certification and Registration Program, in conjunction with the Watershed Management Permits Section, is developing guidance to more specifically address sample preservation and hold time requirements contained in chs. NR 219 and NR 149, Wis. Adm. Code.

- **COMMENT:** There should be some clarification under the information given in the maximum holding time column for parameter 45, orthophosphate, in table F.
RESPONSE: There are nine different forms of orthophosphorus identified in EPA Method 365.1. The requirement for filtration applies only to the determination of dissolved phosphorus species. The department agrees this content is confusing and clarified the intent with the addition of footnote 22, which reads: "Filtration is only required when reporting dissolved orthophosphate, dissolved hydrolyzable phosphorus or dissolved organic phosphorus as described in EPA Method 365.1 (1993). Filtration must be completed within 15 minutes of collection using a $0.45\mu\text{m}$ filter; sample shall be maintained at $\leq 6^{\circ}\text{C}$ and analyzed within 48 hours."

Other Comments

- **COMMENT:** There is only one methodology which is approved for the analysis of mercury in sludge samples, Cold Vapor Atomic Absorption Spectroscopy (CVAAS). EPA SW-846 Method 7474, which is specifically approved for the analysis of sediment and tissue samples uses a combination of microwave digestion, Bromide/Bromate oxidation and Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS) for the determination of total mercury. We are providing data to substantiate the ability to recover mercury from a standard reference material certified by NIST. Please consider inclusion of this method for the analysis of mercury in sludge by CVAFS.
RESPONSE: The department agrees that the fluorescence technique may be appropriate for sludges regulated under this chapter. The rigorous microwave digestion technique described in SW-846 Method 7474, when combined with fluorescence detection will result in data of similar quality to other currently approved analytical methods. Table EM has been modified to incorporate this addition.
- **COMMENT:** Analytical methods contained in *Standard Methods*, 18th, 19th, 20th, and 21st editions refer to sampling and handling preservation temperatures of 4°C . The temperatures listed in Table F, Required Containers, Preservation Techniques, and Holding Times lists $\leq 6^{\circ}\text{C}$. To avoid confusion between the analytical methods and Table F, we propose the following language be incorporated or referenced in NR 219.04: "Sample preservation procedures, container materials, and maximum allowable holding times for parameters care cited in Table F. Information in the table takes precedence over information in specific methods or elsewhere. Any person may apply for a variance from the prescribes preservation techniques, container materials and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the..."
RESPONSE: The hierarchy for sample preservation for laboratories performing analyses for covered programs, including permitted wastewater discharges under, ch. NR 219, Wis. Adm. Code, is contained in the Laboratory Certification and Registration code chapter, specifically s. NR 149.46(4)(a), Wis. Adm. Code, which states: "Laboratories shall follow the sample preservation procedures and holding times required by state and federal regulations. If the sample preservation procedures are not required by state or federal regulations, laboratories shall follow the sample preservation procedures and holding times established in the analytical method. If the analytical method does not establish sample preservation procedures and holding times, laboratories shall follow the procedures in authoritative sources specified in Appendix III of this chapter."

This language means that laboratories must follow all sample preservation and holding times included in state regulations, including ch. NR 219, Table F, Wis. Adm. Code, or 40 CFR Part 136 Table II. The sample preservation and holding time language contained in approved methods in ch. NR 219, Wis. Adm. Code, for example, is applicable only when there are no preservation or hold time requirements established in state or federal regulations. The sample preservation and holding time requirements contained in authoritative sources, such as *Standard Methods for the Analysis of Water and Wastewater*, 20th ed., are applicable only if there are no requirements in state or federal regulations or in approved methods for analyses.

The language contained in the Laboratory Certification and Registration Program code clearly dictates the hierarchy for sample preservation and holding time requirements. The department does not believe that inclusion of the suggested language is necessary as the hierarchy is addressed elsewhere in Wisconsin Administrative Code.

Modifications Made

Modifications made by the department are detailed in the response to comments.

Appearances at the Public Hearing

August 27, 2008 – Madison

In support:

Sharon Mertens, 250 West Seeboth Street, Milwaukee, WI 53204

In opposition- none

As interest may appear- none

August 27, 2008 – Stevens Point

In support- none

In opposition- none

As interest may appear- none

Changes to Rule Analysis and Fiscal Estimate

No modifications were made to the rules analysis or fiscal estimate as a result of public comments.

Response to Legislative Council Rules Clearinghouse Report

The Legislative Council Rules Clearinghouse Report did not contain any comments or suggestions to modify the content of this chapter.

Final Regulatory Flexibility Analysis

The proposed rule does not have a significant economic impact on a substantial number of small businesses. The small businesses impacted by the proposed rule are commercial laboratories that perform compliance monitoring for WPDES permittees. The vast majority of these laboratories have

previously implemented use of newer analytical methods that were retained in Tables A-EM of this chapter. For most parameters where small businesses may have to update their method references, the Department has maintained at least one method from the current language. There were only four specific techniques eliminated— three of these included mercury-containing reagents for which other alternatives using similar techniques were retained. Hexane extractable materials, an alternative to the freon extraction for oil and grease, has been in use by laboratories for over a decade. All of the small businesses that perform oil and grease determinations currently maintain certification for the hexane method. The Department no longer offers certification for the freon extraction method.

Sample preservation requirements for tests that are typically considered field parameters and are not required to be performed by certified laboratories, were clarified so there is no question as to whether data from these time-sensitive tests is valid. The hold times for hexavalent chromium and polychlorinated biphenyls (PCBs) increased; this will allow small businesses more flexibility in scheduling and performing these analyses. Clarification of procedures for potential interferences in cyanide analyses will allow small business laboratories to continue to perform these analyses in 14 days without requiring investment in new instrumentation required to conduct these analyses.

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

The Wisconsin Natural Resources Board proposes an order to amend s. NR 219.04 (2) and (3) (intro), to repeal and recreate NR 210.04 Tables A, B, C, D, E, EM and F; and to create s. NR 219.04 (4) relating to analytical methods used for Wisconsin Pollutant Discharge Monitoring System (WPDES) compliance monitoring.

SS-16-08

Summary Prepared by the Department of Natural Resources

I. Statutes Interpreted

Sections 283.31, 283.55 (1), 299.11 and 299.15(2), Stats.

2. Statutory Authority

Sections 227.11 (2), 281.19(1), 283.31, 283.55(1), 299.11, and 299.15(2), Stats.

3. Explanation of Agency Authority

Section 281.19(1), Stats. authorizes the department to issue general orders, and adopt rules applicable throughout the state for the construction, installation, use and operation of systems, methods and means to prevent and abate pollution of the waters of the state. Section 283.31, Stats. prohibits the discharge of any pollutant into any waters of the state or the disposal of sludge from a treatment work by any person unless such discharge or disposal is done under a permit issued by the department. Section 283.55(1), Stats. requires permittees to use monitoring methods, including where appropriate, biological monitoring methods, to identify and determine the amount of each pollutant discharged from each point source under the owner's or operator's ownership or control. Section 299.15(2)(a), Stats. authorizes the department by rule to prescribe methods of analysis for pollutants.

4. Related Statute or Rule

Chs. NR 101, 102, 106, 149, 157, 200, 204, 205, 206, 210, 211, 212, 214, 216, 217, 218, 230, 233, 243, 252, 254, 256, 260, 261, 262, 263, 270, 273, 274, 279, 290, and 347, Wis. Adm. Code and Chapter 283, Stats.

5. Plain Language Rule Analysis

The analytical methods contained in 40 CFR Part 136, which are used by laboratories in support of WPDES compliance monitoring, have not been substantively updated in several years. Many of the methods that were deleted by EPA were originally published twenty or more years ago. Use of newer laboratory instruments and methods have been hindered by the lack of inclusion in the Federal rule. In March of 2007, EPA published updated analytical test methods. The proposed rule revisions to chapter NR 219 incorporate these updated methods for the WPDES permit program.

The department is also incorporating methods that have been approved through the Office of Water's Alternative Test Procedure program, including the luminescence technique for dissolved oxygen, and selected methods from the 21st Edition of "Standard Methods for the Examination of Water and Wastewater". The tables also include updated references from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Updates III and IV", the "2007 Annual Book of Standards" from ASTM and developed by instrument manufacturers.

The maximum holding time requirement for some tests, as listed in Table F, has been clarified. The current language, "analyze immediately", has been interpreted to mean within 15 minutes or less of sample collection. This has been clarified in the federal rule so that the holding time for these tests is now "analyze within 15 minutes". Other changes to this table include identification of holding times for the various matrices that require dioxins and furan analysis, and allowing metals samples to be transported to a laboratory without pH adjustment. Preservation requirements for available and total cyanide have been expanded to describe procedures that are recommended for removal or suppression of known cyanide interferences.

This proposal also changes language in ss. NR 219.04 (2) and (3) to make this language consistent with the thermal preservation requirements contained in Table F.

6. Summary of, and Comparison with, Existing or Proposed Federal Regulations

The federal counterpart to this rule is 40 CFR 136. On March 12, 2007, EPA published "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule" In addition, on March 26, 2007, EPA published the "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for Biological Pollutants in Wastewater and Sewage Sludge; Final Rule." The proposed revision to ch. NR 219 incorporates changes and are consistent with the federal regulations.

7. Comparison with Rules in Adjacent States

The States of Illinois, Iowa, Michigan, and Minnesota have wastewater programs delegated to them from the U.S. Environmental Protection Agency. The Illinois laboratory certification program required laboratories to update their scope of accreditation to incorporate the revisions starting June 1, 2007. The IL Environmental Protection Agency did not require rulemaking to address these updates. The Iowa Department of Natural Resources incorporated the changes at 40 CFR 136 in rulemaking initiated in November 2007; the changes were effective January 9, 2008. The State of Michigan does not certify laboratories that perform analyses of wastewater. The Minnesota laboratory certification program allowed laboratories to use the analytical methods listed in the federal rules on the effective dates, April 11 and 25, 2007, respectively. Minnesota's program limits analytical methods to those from the 20th and 21st Editions and on-line version of "Standard Methods for the Examination of Water and Wastewater", and limits ASTM methods to the most-recently published version. All other sources of analytical methods were incorporated as described in the federal rule. MN Department of Public Health did not require rulemaking to address these updates.

8. Summary of Factual Data and Analytical Methodologies

This proposed rule revision brings Wisconsin up to date with the current federal rules that establish analytical test methods.

9. Analysis and Supporting Documents Used to Determine Effect on Small Business or in Preparation of Economic Impact Report

Many of the analytical methods that are being deleted have been replaced with methods that are nearly identical. The quality control requirements for analyses are established in ch. NR 149; these will not change with updating methods tables. For several analyses, newer methods and techniques have been added. It is possible for a laboratory to change the analytical method it uses and not incur additional costs or, in some cases, actually reduce costs.

The requirements imposed upon small business include following approved analytical methods listed in the rule. There are no reporting requirements in NR 219. The small businesses that will likely be impacted by this rule are commercial laboratories certified under ch. NR 149. The vast majority of these laboratories

likely follow approved analytical methods that are being maintained in this chapter. The certification status for each small business that was certified for oil and grease was examined to determine whether they performed the deleted and retained methods. Only one laboratory would have to change their certification status. The impacted laboratory has since obtained certification for the retained method for oil and grease determinations.

This proposal does not change the frequency of analytical testing, nor does it address any change in reporting, schedule or deadline requirements. Consequently, the impacts to small businesses will be minimal.

10. Effect on Small Business

The proposed changes to ch. NR 219 potentially affect all certified and registered laboratories and wastewater facilities that collect samples for compliance with their Wisconsin Pollutant Discharge Elimination System (WPDES) permit. There are 420 laboratories certified or registered to perform analyses by the department. Of these, 264 municipal wastewater laboratories, 60 industrial environmental, and 78 commercial environmental will be impacted by this rule. The remaining laboratories are public health laboratories that analyze drinking water and those that perform testing at hazardous waste facilities. This update will allow laboratories to utilize new techniques that currently require approval by the U. S. Environmental Protection Agency and deletes outdated methods that lack adequate quality control procedures.

Of the approximately 400 certified or registered laboratories that will be impacted by this rule, there are less than 12, 3% of the total, that fit the definition of "small businesses". This proposed rule would affect commercial, industrial, and municipal laboratories that analyze effluent discharges to determine compliance with WPDES permit limitations. All laboratories performing analyses to determine WPDES compliance are expected to keep detailed records of all laboratory information, including: analytical methods and results, corrective action logs, equipment records, quality control records, and sample receipt logbooks.

This proposal does not change analytical testing requirements, nor does it address any change in reporting, schedule or deadline requirements. Sections 299.11 and 283.55, Stats., do not allow for less stringent schedules, deadlines or reporting requirements. Laboratories may seek approval of an alternate test procedure, as specified in s. NR 219.05, Wisc. Adm. Code, from U.S. EPA. WPDES permit terms and conditions and other related regulations require that permitted facilities conduct analytical tests in compliance with the procedures of ch. NR 219. The Laboratory Certification and Registration Program audits laboratories performing these analyses to ensure that testing is performed in compliance with the proposed rule. If a laboratory does not follow an approved method, it is cited during the audit process; a laboratory must correct all identified deficiencies to maintain their certification under ch. NR 149.

11. Anticipated Costs Incurred by Private Sector

The vast majority of laboratories will not incur additional costs as a result of this rulemaking. The replacement of deleted methods with similar, and in some cases, identical language, does not require laboratories to incur additional costs. The laboratory community was advised of EPA's intent to delete analytical methods when the draft rule was published in 2003. In 2004 revisions to chapter NR 219, Wis. Adm. Code, the department incorporated many updated methods from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846".

Many of the laboratories that perform oil and grease determinations have maintained certifications for both the freon and hexane extractable materials techniques. The use of freon as an extraction solvent for oil and grease determinations, was eliminated by EPA in 2001. Since that time, the department has worked to modify permit analytical requirements to utilize the hexane extractable material method and eliminate the use of freon. Those laboratories that perform oil and grease determinations will see a cost-savings by the elimination of the freon technique. One laboratory noted a 60-pound container of freon cost approximately

\$1,400; the same volume of hexane costs approximately \$140. Utilization of hexane instead of freon as an extraction solvent for oil and grease results in significant cost-savings for laboratories.

12. Agency Contact Person

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Section 1: NR 219.04 (2) and (3)(intro) are amended to read:

(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 less than or equal to 6° C where required. All samples requiring thermal preservation at 4 less than or equal to 6° 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 will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

(3) TEMPERATURE REPORTING PROCEDURES. (intro) Samples cooled with ice packs or not in direct contact with ice during shipping shall be cooled to -4 less than or equal to 6° 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:

Section 2: NR 219.04 (4) is created to read:

(4). INCORPORATION BY REFERENCE. The materials in this section are incorporated by reference for the purposes of the permit program under chapter 283, Stats.

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 and the legislative reference bureau.~~ Many of these materials are also available through inter-library loan.

Section 3: NR 219.04 Table A is repealed and recreated to read:

Table A
List of Approved Biological Analytical Methods

Parameter and Units	Analytical Technology ¹	EPA	Standard Methods ⁴	Standard Methods Online ¹²	AOAC, ASTM, USGS	Other
Bacteria:						
1. Coliform (fecal), number per 100 mL, or number per gram dry weight	Most Probable Number (MPN), 5 tube 3 dilution, or Membrane filter (MF) ³ , single step	p. 132 ³ , 1680 ¹² , 1681 ^{12, 19} , 1684 ³	9221 C or E [18 th , 19 th , 20 th , 21 st] 9222 D [18 th , 19 th , 20 th , 21 st]	9221 C-99 or 9221 E-99 9222 D-97	B-0050-85 ⁵ 991.15 ¹¹	Colilert [®] 13, 17, Colilert-18 [®] 13, 16, 17
2. Coliform (fecal) in presence of chlorine, number per 100 mL	MPN, multiple tube/multiple well	p. 132 ³	9221 C or E [18 th , 19 th , 20 th , 21 st]	9221 C-99 or 9221 E-99		
3. Coliform (total), in number per 100 mL	MPN, multiple tube/multiple well, or MF ² , single step	p. 124 ³	9223 B [18 th , 19 th , 20 th , 21 st] 9222 D [18 th , 19 th , 20 th , 21 st]	9223 B-97 9222 D-97	991.15 ¹¹	Colilert [®] 13, 17, Colilert-18 [®] 13, 16, 17
4. Coliform (total), in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or MF ² , single step or two step	p. 114 ³ p. 108 ³	9221 B [18 th , 19 th , 20 th , 21 st] 9222 B [18 th , 19 th , 20 th , 21 st]	9221 B-99 9222 B-97	B-0025-85 ⁵	
5. <i>E. coli</i> , number per 100 mL ²⁰	MPN, 5 tube, 3 dilution, or MF ² with enrichment MPN ^{7, 15} , multiple tube	p. 114 ³ p. 111 ³	9221 B [18 th , 19 th , 20 th , 21 st] 9222 (B+B, 5c) [18 th , 19 th , 20 th , 21 st] 9221 B.1 or 9221 F [18 th , 19 th , 20 th , 21 st , 25 th]	9221 B-99 9222 (B+B, 5c)-97 9221 B.1 or F-99 ^{22, 25}		
6. Fecal streptococci, number per 100 mL	MPN, multiple tube/multiple well	p. 1103.1 ²⁷ 1603 ²¹ , 1604 ²⁸	9223 B [18 th , 19 th , 20 th , 21 st] ¹³ 9222 B [18 th , 19 th , 20 th , 21 st] ²⁶ , 9222 G [18 th , 19 th , 20 th] ²⁶ , 9213 D [18 th , 19 th , 20 th]	9223 B-97 ¹³ 9222 B-97 ²⁶ or G-97 ²⁶	991.15 ¹¹ D5392-93 ¹⁰	Colilert [®] 13, 17, Colilert-18 [®] 13, 16, 17
7. Enterococci, number per 100 mL ³⁰	MPN, 5 tube, 3 dilution, or MF ² , or Plate count	p. 139 ³ p. 136 ³ p. 143 ³	9230 B [18 th , 19 th , 20 th , 21 st] 9230 C [18 th , 19 th , 20 th , 21 st]	9230 B-93 9230 C-93	B-0055-85 ⁵	mColiBlue-24 [®] 18
8. Cryptosporidium	MPN, multiple tube/multiple well, or MF ² , two step	1106.1 ²⁹	9230 B [18 th , 19 th , 20 th , 21 st]	9230 B-93		
9. Giardia	MPN, multiple tube/multiple well, or Plate count	1600 ²⁴ p. 143 ³	9230 C [18 th , 19 th , 20 th , 21 st]	9230 C-93	D6503-99 ¹⁰ D5259-92 ¹⁰	Enterolert [®] 13, 23
Aquatic Toxicity:						
10. Toxicity, acute, fresh water organisms, percent effluent mortality	Ceriodaphnia, 48-h static-renewal mortality	1622 ³⁰ , 1623 ³¹ 1623 ³¹				Note 32

Parameter and Units	Analytical Technology ¹	EPA	Standard Methods ⁴	Standard Methods Online ¹²	AOAC, ASTM, USGS	Other
11 Toxicity, chronic, fresh water organisms, percent effluent	Fathead minnow, 96-h static renewal mortality, or 96-h flow-through mortality Ceriodaphnia survival and reproduction Fathead minnow larval survival and growth					Note 32 Note 32 Note 32

¹ The method must be specified when results are reported.

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

³ U.S. EPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Protection Agency, Cincinnati, OH, EPA/600/8-78/017.

⁴ 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, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

⁵ USGS. 1989. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, VA.

⁶ Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

⁷ Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

⁸ When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

⁹ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

¹⁰ ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

¹¹ AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume 1, Chapter 17. Association of Official Analytical Chemists International, 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877-2417.

¹² Standard Methods for the Examination of Water and Wastewater On-Line, Joint Editorial Board, American Public Health Association, American Water Works Association, Water Environment Federation, 2006. Subscription service available at: <http://www.standardmethods.org>.

¹³ These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme b-galactosidase produced by *E. coli*.

¹⁴ U.S. EPA. July 2006. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (L.TB) and EC Medium. US Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-012.

¹⁵ Samples shall be enumerated by the multiple-tube or multiple-tube procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert[®] may be enumerated with the multiple-well procedures, Quanti-Tray[®] 2000, and the MPN calculated from the table provided by the manufacturer.

¹⁶ Colilert-18[®] is an optimized formulation of the Colilert[®] for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C rather than the 24 h required for the Colilert[®] test and is recommended for marine water samples.

¹⁷ Descriptions of the Colilert[®], Colilert-18[®], Quanti-Tray[®], and Quanti-Tray[®] 2000 may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

¹⁸ A description of the mColiBlue24[®] test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

¹⁹ U.S. EPA. July 2006. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-013.

²⁰ Recommended for enumeration of target organism in wastewater effluent.

²¹ U.S. EPA. July 2006. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-011.

²² The multiple-tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (L.TB), if at least 25 parallel tests are conducted between this broth and L.TB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

²³ A description of the Enterolert[®] test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

¹⁴ U.S. EPA, July 2006, Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-b-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-009.

¹⁵ After prior enrichment in a presumptive medium for total coliform using 922 [B.1., all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

¹⁶ Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA-MUG media.

¹⁷ U.S. EPA, July 2006, Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-010.

¹⁸ U.S. EPA, September 2002, Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA 821-R-02-024.

¹⁹ U.S. EPA, July 2006, Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-008.

²⁰ Method 1622 uses filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. U.S. EPA, 2001, Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-01-026.

²¹ Method 1623 uses filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. U.S. EPA, 2001, Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-01-025.

²² Compliance monitoring must be performed in accordance with the specifications in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 2nd Edition," Wisconsin Department of Natural Resources, 2004. This publication is available for inspection at the offices of the Department of Natural Resources and the Legislative Reference Bureau. Copies are available from the Department of Natural Resources, Bureau of Science Services, P.O. Box 7921, Madison, WI 53707.

Section 4: NR 219.04 Table B is repealed and recreated to read:

Table B
List of Approved Inorganic Analytical Methods in Wastewater

Parameter, Units	Analytical Technology ¹⁸	EPA ^{14, 16}	SW-846 ¹⁵	Standard Methods [Editions] ¹⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
1. Acidity, as CaCO ₃ , mg/L;	Electrometric or phenolphthalein endpoint			2310 B(4a) [18 th , 19 th , 20 th , 21 st]	2310 B(4a)-97	D1067-92, 02, 06	I-1020-85 ²	
2. Alkalinity, as CaCO ₃ , mg/L;	Electrometric or colorimetric titration to pH 4.5, manual, or automatic	310 2, (Rev. 1974) ¹		2320 B [18 th , 19 th , 20 th , 21 st]	2320 B-97	D1067-92, 02, 06	I-1030-85 ²	973, 43 ¹
3. Aluminum- Total ⁹ , mg/L;	Digestion ^{9, 11, 15} followed by:						I-2030-85 ²	
	AA direct aspiration (FLAA)		7000B	3111 D [18 th , 19 th , 21 st]	3111 D-99			
	AA graphic furnace (GF-AA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99		I-3051-85 ²	

Parameter, Units	Analytical Technology ¹⁸	EPA ^{44, 42}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120B [21 st]	3120 B-99		1-4471-97 ²	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ³
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Orthochrome cyanine R)			3500-AI D [18 th , 19 th], 3500 AI B [20 th , 21 st]	3500-AI B-01			
4. Ammonia (as N), mg/L:	Manual distillation (at pH 9.5) followed by:	350 L, Rev. 2.0 (1993)		4500- NH ₃ B [18 th , 19 th , 20 th , 21 st]	4500- NH ₃ B-97			973.49 ³
	Titration			4500- NH ₃ E [18 th], 4500- NH ₃ C [19 th , 20 th , 21 st]	4500- NH ₃ -C-97			
	Electrode			4500- NH ₃ F or G [18 th], 4500- NH ₃ D or E [19 th , 20 th , 21 st]	4500- NH ₃ D or E-97	D1426-98, 03 (B)		
	Automated phenate	350 L, Rev. 2.0, (1993) ^{7b}		4500- NH ₃ H [18 th], 4500- NH ₃ G [19 th , 20 th , 21 st]	4500- NH ₃ G-97		1-4523--85 ²	
	Automated electrode, or ion chromatography					D6919-03		Note 15
5. Ammony- Total ⁹ , ug/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99			
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99			
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ³
6. Arsenic- Total ⁹ , ug/L:	Digestion ^{9, 11, 45} followed by:							
	AA gaseous hydride		7061A	3114 B ¹⁰ [18 th , 19 th , 21 st]	3114 B 4.d.-97	D2972-97, 03 (B)	1-3062-85 ²	

Parameter, Units	Analytical Technology ⁶⁸	EPA ^{4, 52}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
7. Barium- Total ⁹ , mg/L:	AA graphite furnace (GF-AA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D2972-97, 03 (C)	1-4063-98 ⁹⁹	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Colorimetric (SDCC)			3500-As C [18 th , 19 th , 21 st], 3500-As B [20 th , 21 st]	3500-As B-97	D2972-97, 03(A)	1-3060-85 ²	
	Digestion ^{11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 th , 19 th , 21 st]	3111 D-99		1-3084-85 ²	
	AA graphite furnace (GF-AA)		7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D4382-95, 02		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Direct current plasma (DCP)							Note 43
8. Beryllium- Total ⁹ , mg/L:	Digestion ^{11, 45} followed by:							
	AA direct aspiration (FL-AA)		7000B	3111 D [18 th , 19 th , 21 st]	3111 D-99	D3645-93 (88), 03 (A)	1-3095-85 ²	
	AA graphite furnace (GF-AA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D3645-93 (88), 03 (B)		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		1-4471-97 ¹⁰⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Aluminum)			3500-Bc D [18 th , 19 th]				

Parameter, Units	Analytical Technology ^{10,11}	EPA ^{14, 15}	SW-846 ^{1, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
9. Biochemical Oxygen Demand (BOD ₅), mg/L;	Dissolved oxygen depletion			5210 B [18 th , 19 th , 20 th , 21 st]	5210 B-01		I-1578-78 ¹⁶	973.443 ¹ , p17 ¹⁶
10. Boron ⁴ , mg/L;	Colorimetric (Curcumin)			4500-B B [18 th , 19 th , 20 th , 21 st]	4500-B B-00		I-3112-85 ²	
	Inductively coupled plasma-atomic emission spectrometry (ICP) ^{11, 15}	200.7, Rev. 4.4 (1994) ¹⁵	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		I-4471-97 ¹⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹⁵	6020A					
	Direct current plasma (DCP) ^{11, 15}					D4190-94, 99, 03		Note 43
11. Bromide, mg/L;	Titrimetric					D1246-95, 99 (C)	I-1125-85 ²	p. S44 ¹⁸
		300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		993.30 ³
	CIE/UV					D6508-00 (05)		D6508, Rev 2 ¹⁴
12. Cadmium- Total ⁹ mg/L;	Digestion ^{9, 11, 15} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99	D3557-95, 02 (A or B)	I-3135-85 ² or I-3136-85 ²	974.27 ³ , p. 37 ¹⁷
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹⁵	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D3557-95, 02 (D)	I-4138-89 ¹¹	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹⁵	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		I-1472-85 ² or I-4471-97 ¹⁰	993.14 ¹
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹⁵	6020A			D5673-03, 05		
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Voltammetry ¹⁹ , or					D3557-95, 02 (C)		
	Colorimetric (Dithizone)			3500-Cd D [18 th , 19 th]				

Parameter, Units	Analytical Technology ^{6a}	EPA ^{4, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁴	USGS	Other
13. Calcium- Total ⁸ , mg/L:	Digestion ^{9, 11, 15} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99	D511-93, 03 (B)	I-3152-85 ²	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		I-4471-97 ⁶⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹⁵	6020A					
	Direct current plasma (DCP)							Note 43
	Titrimetric (EDTA), or			3500-Ca D [18 th , 19 th , 20 th , 21 st]	3500-Ca B-97	D511-93, 03 (A)		
	Ion chromatography					D6919-03		
14. Carbonaceous Biochemical Oxygen Demand (CBOD ₅) ²⁰ , mg/L:	Dissolved oxygen depletion with nitrification inhibitor			5210 B [18 th , 19 th , 20 th , 21 st]	5210 B-01			
15. Chemical Oxygen Demand (COD), mg/L:	Titrimetric, or	410.3, (Rev. 1978) ¹		5220 C [18 th , 19 th , 20 th , 21 st]	5220 C-97	D-1252-95, 00, 06 (A)	I-3560-85 ²	973.46 ¹ and p.17 ¹⁷
	Spectrophotometric, manual or automatic	410.4, Rev. 2.0 (1993)		5520 D [18 th , 19 th , 20 th , 21 st]	5220 D-97	D1252-95, 00, 06 (B)	I-3561-85 ²	Notes 21, 22
16. Chloride, mg/L:	Titrimetric (Silver nitrate) or		9253	4500-Cl B [18 th , 19 th , 20 th , 21 st]	4500-Cl B-97	D512-89 (99), 04 (B)	I-1183-85 ²	
	Colorimetric; manual or automated (Ferricyanide)		9250	4500-Cl E [18 th , 19 th , 20 th , 21 st]			I-1187-85 ²	
	Potentiometric titration			4500-Cl D [18 th , 19 th , 20 th , 21 st]	4500-Cl E-97		I-2187-85 ²	
	Ion selective electrode				4500-Cl D-97			
		300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)				D512-89 (99), 04 (C)		
	Ion chromatography, or		9056	4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		993.30 ¹
	CIE/UV					D6508-00 (05)		D6508, Rev. 2 ^(a)

Parameter, Units	Analytical Technology ⁸⁸	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁴	USGS	Other
17. Chlorine, Total Residual, mg/L	Amperometric direct			4500-Cl D [18 th , 19 th , 20 th , 21 st]	4500-Cl D-00	D1253-86 (96), 03		
	Amperometric direct (low level)			4500-Cl E [18 th , 19 th , 20 th , 21 st]	4500-Cl E-00			
	Iodometric direct			4500-Cl B [18 th , 19 th , 20 th , 21 st]	4500-Cl B-00			
	Back titration either endpoint ⁴¹			4500-Cl C [18 th , 19 th , 20 th , 21 st]	4500-Cl C-00			
	DPD-FAS			4500-Cl F [18 th , 19 th , 20 th , 21 st]	4500-Cl F-00			
	Spectrophotometric, DPD, or			4500-Cl G [18 th , 19 th , 20 th , 21 st]	4500-Cl G-00			Note 24
	Ion selective electrode							
18. Chromium VI, dissolved, ug/L	0.45 micron filtration followed by:							
	AA chelation-extraction	218.6, Rev. 3.3 (1994)	7197	3111 C [18 th , 19 th , 21 st]	3111 C-99		1-1232-85 ²	
	Ion chromatography, or		7199	3500-Cr E [18 th , 19 th], 3500-Cr C [20 th , 21 st]	3500-Cr C-01	D5257-97, 03		993.23 ¹
	Colorimetric (Diphenylcarbazide)			3500-Cr D [18 th , 19 th], 3500-Cr B [20 th , 21 st]	3500-Cr B-01	D1687-92, 02 (A)	1-1230-85 ²	
19. Chromium- Total ⁹ , mg/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99	D1687-92, 02 (B)	1-3236-85 ²	974.27 ¹
	AA chelation-extraction			3111 C [18 th , 19 th , 21 st]	3111 C-99			
	AA graphite furnace (GF-AA)	200.9, Rev. 2.2 (1994) ¹⁵	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D1687-92, 02 (C)	1-3233-93 ⁵⁶	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹⁵	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹⁵	6020A			D5673-03, 05		993.14 ³
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Diphenylcarbazide)			3500-Cr D [18 th , 19 th], 3500-Cr B [20 th , 21 st]	3500-Cr B-01			

Parameter, Units	Analytical Technology ⁶⁸	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
20. Cobalt- Total ⁹ , mg/L:	Digestion ^{11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99	D3558-94, 03 (A or B)	I-3239-85 ³	p 37 ¹⁷
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D3558-94, 03 (C)	I-4243-89 ⁶¹	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		I-4471-97 ⁶⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ³
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
21. Color, platinum cobalt units or dominant wavelength, hue, luminance, purity:	Colorimetric (ADMI)			2120 E [18 th , 19 th , 20 th , 21 st]				Note 26
	(Platinum cobalt), or			2120 B [18 th , 19 th , 20 th , 21 st]	2120 B-01		I-1250-85 ²	
	Spectrophotometric			2120 C [18 th , 19 th , 20 th , 21 st]				
22. Copper- Total ⁹ , mg/L:	Digestion ^{11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99	D1688-95, 02 (A or B)	I-3270-85 ² or I3271-85 ³	974.37 ³ , p 37 ¹⁷
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D1688-95, 02 (C)	I-4274-89 ⁶¹	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		I-4471-97 ⁶⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ³
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Colorimetric (Neocuproine), or			3500-Cu D [18 th , 19 th], 3500-Cu B [20 th , 21 st]	3500-Cu B-99			
	(Bicinchoninate)			3500-Cu E [18 th , 19 th], 3500-Cu C [20 th , 21 st]	3500-Cu C-99			Note 27

Parameter, Units	Analytical Technology ¹⁸	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
23. Cyanide, Total, ug/L:	Automated Distillation and Colorimetry							Kelada-01 ⁶⁵
	Manual distillation with MgCl ₂ followed by: Titrimetric	335.4, Rev. 1.0 (1993) ⁶⁶	9010B, 9010C 9014	4500-CN-C [18 th , 19 th , 20 th , 21 st] 4500-CN-D [18 th , 19 th , 20 th , 21 st]	4500-CN-D-99	D2036-98, 06(A)		10-204-00-1- X ⁶⁶ p.22 ¹⁷
	Spectrophotometric, manual		9014	4500-CN-E [18 th , 19 th , 20 th , 21 st]	4500-CN-E-99	D2036-98, 06 (A)	1-3300-85 ²	
	Automated ⁶⁸ , or Ion selective electrode	335.4, Rev. 1.0 (1993) ⁶⁷	9012A, 9012B	4500-CN-F [18 th , 19 th , 20 th , 21 st]	4500-CN-F-99	D2036-98, 06 (A)	1-4302-85 ²	10-204-00-1- X ⁶⁶
24. Cyanide, Available, ug/L:	Cyanide Amenable to Chlorination (CATC), Manual distillation with MgCl ₂ followed by Titrimetric or Spectrophotometric		9010B, 9010C, 9014	4500-CN-G [18 th , 19 th , 20 th , 21 st]	4500-CN-G-99	D2036-98, 06 (B)		
	Flow injection and ligand exchange, followed by amperometry ⁷¹ , or Automated distillation and colorimetry		9012A			D6888-04		OIA-1677 ⁵⁴
25. Fluoride- Total, mg/L:	Manual distillation ¹⁴ followed by: Electrode, manual Automated Colorimetric (SPADNS) Automated complexone			4500-F-B [18 th , 19 th , 20 th , 21 st] 4500-F-C [18 th , 19 th , 20 th , 21 st] 4500-F-D [18 th , 19 th , 20 th , 21 st] 4500-F-E [18 th , 19 th , 20 th , 21 st]	4500-F-B-97 4500-F-C-97 4500-F-D-97 4500-F-E-97	D1179-93, 99, 04 (B) D1179-93, 99, 04 (A)	1-4327-85 ²	
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		993.30 ³

Parameter, Units	Analytical Technology ⁶⁸	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
26. Gold- Total ⁹ , mg/L:	CIE/UV					D6508-00 (05)		D6508, Rev. 2 ⁶⁴
	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99			
	AA graphite furnace (GFAA)	231.2, Rev. 1978 ¹						
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A					
	Direct current plasma (DCP)							Note 43
27. Hardness, Total as CaCO ₃ , mg/L:	Automated colorimetric	130.1, (Issued 1971) ¹						
	Titrimetric (EDTA), or			2340 B or C [18 th , 19 th , 20 th , 21 st]	2340 B or C-97	D1126-86 (92), 02	1-1338-85 ²	973.52B ¹
	Ca plus Mg as their carbonates by inductively coupled plasma-atomic emission spectrometry (ICP) or							
	AA direct aspiration (See Parameters 13 and 33)							
28. Hydrogen ion (pH), pH units:	Electrometric measurement or		9040C	4500-H ⁺ B [18 th , 19 th , 20 th , 21 st]	4500-H ⁺ B-00	D1293-84 (90), 99 (05) (A or B)	1-1586-85 ²	973.41 ¹
	Automated electrode	150.2, (Dec. 1982) ¹					1-2587-85 ²	Note 29
29. Iridium- Total ⁹ , ug/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)			3111 B [18 th , 19 th , 21 st]	3111 B-99			
	AA graphite furnace (GFAA)	235.2, (Issued 1978) ¹	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			

Parameter, Units	Analytical Technology ^{6a}	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ^{6b}	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A					
30. Iron- Total, mg/L	Digestion ^{3, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99	D1068-96, 03, 05 (A or B)	1-3381-85 ²	974.27 ¹
	AA graphite furnace (GF-AA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D1068-96, 03, 05 (C)	1-4471-97 ^{6a}	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A					Note 43
	Direct current plasma (DCP), or							Note 30
	Colorimetric (Phenanthroline)			3500-Fe D [18 th , 19 th], 3500-Fe B [20 th , 21 st]	3500-Fe B-97	D4190-94, 99, 03		
31. Kjeldahl Nitrogen- Total ¹² , (as N), mg/l	Digestion and distillation ^{3a} followed by:			4500-N _{org} B or C [18 th , 19 th , 20 th , 21 st] and 4500-NH ₃ B [18 th , 19 th , 20 th , 21 st]	4500-N _{org} B or C-97 and 4500-NH ₃ B-97	D3590-89, 02 (06) (A)		
	Titration			4500-NH ₃ E [18 th], 4500-NH ₃ C [19 th , 20 th , 21 st]	4500-NH ₃ C-97	D3590-89, 02 (06) (A)		973.48 ¹
	Electrode			4500-NH ₃ F or G [18 th], 4500-NH ₃ D or E [19 th , 20 th , 21 st]	4500-NH ₃ D or E-97			
	Automated phenate colorimetric	351.1, (Rev. 1978) ¹					1-4551-78 ¹⁶	
	Semi-automated block digester colorimetric	351.2, Rev. 2.0 (1993)				D3590-89, 02 (06) (B)	1-4515-91 ¹⁵	
	Manual or block digester potentiometric					D3590-89, 02 (06) (A)		
	Block digester, followed by auto distillation and titration, or							Note 48

Parameter, Units	Analytical Technology ^{6a}	EPA ^{11, 62}	SW-846 ^{1, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
32. Lead- Total ⁶ , mg/L:	Flow injection gas diffusion Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) AA graphite furnace (GFAA) Inductively coupled plasma-atomic emission spectrometry (ICP) Inductively coupled plasma-mass spectrometry (ICP-MS) Direct current plasma (DCP) Voltammetry, or Colorimetric (Diithizone)	200.9, Rev. 2.2 (1994) ¹¹ 200.7, Rev. 4.4 (1994) ¹³ 200.8, Rev. 5.4 (1994) ¹³	7000B 7010 6010B, 6010C 6020A	3111 B or C [18 th , 19 th , 21 st] 3113 B [18 th , 19 th , 21 st] 3120 B [20 th , 21 st] 3500-Pb B [20 th , 21 st] 3500-Pb D [18 th , 19 th , 3500-Pb B [20 th , 21 st]	3111 B or C-99 3113 B-99 3120 B-99 3500-Pb B-97	D3559-96, 03 (A or B) D3559-96, 03 (D) D5673-03, 05 D4190-94, 99, 03 D3559-96, 03 (C)	1-3399-90 ² 1-4403-89 ⁶¹ 1-4471-97 ⁶⁰	Note 49 974.27 ¹ 974.27 ¹
33. Magnesium- Total ⁶ , mg/L:	Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) Inductively coupled plasma-atomic emission spectrometry (ICP) Inductively coupled plasma-mass spectrometry (ICP-MS) Direct current plasma (DCP) Gravimetric, or Ion chromatography	200.7, Rev. 4.4 (1994) ¹³ 200.8, Rev. 5.4 (1994) ¹³	7000B 6010B, 6010C 6020A	3111 B [18 th , 19 th , 21 st] 3120 B [20 th , 21 st] 3500-Mg D [18 th , 19 th]	3111 B-99 3120 B-99 D6919-03	D511-93, 03 (B) D858-95, 02 (A or B) D858-95, 02 (C)	1-3447-85 ² 1-4471-97 ⁶⁰	Note 43 974.27 ¹ 974.27 ¹
34. Manganese- Total ⁶ , mg/L:	Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³	7000B 7010	3111 B [18 th , 19 th , 21 st] 3113 B [18 th , 19 th , 21 st]	3111 B-99 3113 B-99	D858-95, 02 (A or B) D858-95, 02 (C)	1-3454-85 ²	974.27 ¹

Parameter, Units	Analytical Technology ^{6a}	EPA ^{41,62}	SW-846 ^{4,5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		1-4471-97 ^{6a}	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Direct current plasma (DCP)				3500-Mn B-99	D4190-94, 99, 03		Note 43
	Colorimetric (Persulfate), or (Periodate)			3500-Mn D [18 th , 19 th , 3500-Mn B [20 th , 21 st]				920.203 ¹
35. Mercury- Total ⁹ , ug/L:	Cold vapor, manual or Automated	245.1	7470A	3112 B [18 th , 19 th , 21 st]	3112 B-99	D3223-97, 02	1-3462-85 ²	Note 31 977.22 ¹
	Purge and trap cold vapor atomic fluorescence spectrometry ⁵¹ (CVAFS), or	245.2						
	Cold vapor atomic fluorescence spectrometry (CVAFS) ⁵⁵	245.7 ⁶⁹						
36. Molybdenum- Total ⁹ , mg/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 th , 19 th , 21 st]	3111 D-99		1-3490-85 ²	
	AA graphite furnace (GFAA)		7010	3113 B [18 th , 19 th , 21 st]	3113 B-99		1-3492-96 ⁵⁷	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		1-4471-97 ^{6a}	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Direct current plasma (DCP)							Note 43
37. Nickel- Total ⁹ , mg/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99	D1886-90, 94 (98), 03 (A or B)	1-3499-85 ²	

Parameter, Units	Analytical Technology ¹⁸	EPA ^{14, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹⁵	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99	D1886-90, 94 (98), 03 (C)	I-4503-89 ⁶¹	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹⁵	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		I-4471-97 ⁶⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or Direct current plasma (DCP)	200.8, Rev. 5.4 (1994) ¹⁵	6020A			D5673-03, 05		993.14 ³
38. Nitrate (as N), mg/L;	Nitrate-nitrite N minus Nitrite N					D4190-94, 99, 03		Note 43
	Ion chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)		4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		993.30 ³
	Ion selective electrode, or CIE/UV			4500-NO ₃ D [18 th , 19 th , 20 th , 21 st]	4500-NO ₃ D-00			
39. Nitrate + Nitrite (as N), mg/L;	Cadmium reduction, manual			4500-NO ₃ E [18 th , 19 th , 20 th , 21 st]	4500-NO ₃ E-00	D6508-00 (05)		D6508, Rev 2 ⁶⁴
	Automated	353.2, Rev. 2.0 (1993)		4500-NO ₃ F [18 th , 19 th , 20 th , 21 st]	4500-NO ₃ F-00	D3867-99, 04(B)		
	Automated hydrazine			4500-NO ₃ H [18 th , 19 th , 20 th , 21 st]	4500-NO ₃ H-00	D3867-99, 04(A)	I-4545-85 ²	
	Ion chromatography ¹⁴ , or CIE/UV	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 th , 21 st]	4110 B-00	D4327-03		993.30 ³
40. Nitrite (as N), mg/L;	Spectrophotometric, Manual			4500-NO ₂ B [18 th , 19 th , 20 th , 21 st]	4500-NO ₂ B-00	D6508-00 (05)		D6508, Rev 2 ⁶⁴
	Automated (Diazotization)						I-4540-85 ²	Note 33

Parameter Units	Analytical Technology ⁴⁶	EPA ^{44, 42}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993)		4500-NO ₃ F [18 th , 19 th , 20 th , 21 st]	4500-NO ₃ F-00	D3867-99, 04(A)	1-4545-85 ²	
	Manual (*bypass cadmium reduction)	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)		4500-NO ₃ E [18 th , 19 th , 20 th , 21 st]	4500-NO ₃ E-00	D3867-99, 04(B)		993.30 ³
	ion chromatography ³⁴ , or		9056	4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		
	CIE/UV				5520 B-01	D6508-00 (05)		D6508, Rev 2 ⁴⁴
41. Oil and Grease- Total recoverable, mg/L	Hexane extractable material (HEM), or	1664A ⁵⁰						
	Silica-gel treated HEM (SGT-HEM); Silica gel treatment and gravimetry	1664A ⁵⁰						973.47 ¹ , p.14 ¹²
42. Organic Carbon, Total (TOC), mg/L	Combustion or oxidation			5310 B, C or D [18 th , 19 th , 20 th , 21 st]	5310 B, C or D-00			
43. Organic Halides, Adsorbable (AOX), ug/L	Adsorption and coulometric titration	1650 ⁵¹						
44. Organic Nitrogen (as N), mg/L	Kjeldahl nitrogen, total (Parameter 31) minus Ammonia nitrogen (Parameter 4)							
45. Orthophosphate (as P), mg/L	Ascorbic acid method;							
	Automated	365.1, Rev. 2.0 (1993) ¹		4500-P-F [18 th , 19 th , 20 th , 21 st]			1-4601-85 ²	973.56 ³
	Manual single reagent			4500-P-E [18 th , 19 th , 20 th , 21 st]		D515-88(A)		973.55 ³
	Manual two reagent	365.3, (Issued 1978)						
	ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		993.30 ³

Parameter Units	Analytical Technology ^{6,8}	EPA ^{4, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
46. Osmium- Total ⁹ , ug/L:	CHE/UV Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) AA furnace Inductively coupled plasma-atomic emission spectrometry (ICP), or Inductively coupled plasma-mass spectrometry (ICP-MS)	252.2, (Issued 1978) ¹ 200.7, Rev. 4.4 (1994) ¹³ 200.8, Rev. 5.4 (1994) ¹³	7000B 7010 6020A	3111 D [18 th , 19 th , 21 st]	3111 D-99	D6508-00 (05)		D6508, Rev. 2 nd
47. Oxygen, Dissolved, mg/L:	Winkler (azide modification) Electrode, or Luminescence			4500-O C [18 th , 19 th , 20 th , 21 st] 4500-O G [18 th , 19 th , 20 th , 21 st]	4500-O C-01 4500-O G-01	D888-92, 03, 05 (A) D888-92, 03, 05 (B) D888-05(C)	1-1575-78 ¹⁶ 11576-78 ¹⁶	973.45B ¹ Note 72
48. Palladium- Total ⁹ , mg/L:	Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) AA graphite furnace (GFAA) Inductively coupled plasma-atomic emission spectrometry (ICP) Inductively coupled plasma-mass spectrometry (ICP-MS), or Direct current plasma (DCP)	253.2, (Issued 1978) ¹ 200.7, Rev. 4.4 (1994) ¹³ 200.8, Rev. 5.4 (1994) ¹³	7010 6020A	3111 B [18 th , 19 th , 21 st] 3120 B [20 th , 21 st]	3111 B-99 3120 B-99			p. S27 ¹⁸ p. S28 ¹⁸
49. Phenols, ug/L:	Manual distillation ¹⁵ followed by: Colorimetric (4-AAP) manual, or	420.1, (Rev. 1978) ¹ 420.1, (Rev. 1978) ¹	9065					Note 43 Note 36 Note 36

Parameter	Units	Analytical Technology ^{6a}	EPA ^{4, 6a}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
		Automated	420.4, Rev. 1.0 (1993)	9066					Note 37
50. Phosphorus (elemental)	mg/L	Gas-liquid chromatography							973.55 ¹
51. Phosphorus- Total, mg/L		Persulfate digestion ²⁸ followed by: Manual	365.3, (Issued 1978) ¹		4500-P B.5 [18 th , 19 th , 20 th , 21 st]		D515-88(A)	1-4600-85 ²	973.56 ³
		Automated ascorbic acid reduction, or	365.1, Rev. 2.0, (1993)		4500-P E [18 th , 19 th , 20 th , 21 st]				
		Semi-automated block digester	365.4, (Issued 1974) ¹				D515-88(B)	1-4610-91 ³⁸	
52. Platinum- Total ⁹ , mg/L		Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) AA graphite furnace (GF AA)		7010	3111 B [18 th , 19 th , 21 st]	3111 B-99			
		Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³		3120 B [20 th , 21 st]	3120 B-99			
		Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) ¹³	6020A					Note 43
		Direct current plasma (DCP)							
53. Potassium- Total ⁹ , mg/L		Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA) Inductively coupled plasma-atomic emission spectrometry (ICP) Inductively coupled plasma-mass spectrometry (ICP-MS)		7000B 6010B, 6010C 6020A	3111 B [18 th , 19 th , 21 st] 3120 B [20 th , 21 st]	3111 B-99 3120 B-99		1-3630-85 ²	973.53 ¹
		Flame photometric	200.8, Rev. 5.4 (1994) ¹³		3500-K D [18 th , 19 th], 3500-K B [20 th , 21 st]	3500-K B-99			

Parameter	Units	Analytical Technology ⁵⁸	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
		Colorimetric, or ion chromatography					D6919-03		317B ²⁵
54. Residue, total,	mg/L	Gravimetric, 103-105°C			2540 B [18 th , 19 th , 20 th , 21 st]	2540 B-97		1-3750-85 ²	
55. Residue-- filterable, mg/L;		Gravimetric, 180°C			2540 C [18 th , 19 th , 20 th , 21 st]	2540 C-97		1-1750-85 ²	
56. Residue, non-filterable, mg/L;		Gravimetric, 103-105°C post-washing of residue			2540 D [18 th , 19 th , 20 th , 21 st]	2540 D-97		1-3765-85 ²	
57. Residue, settleable, mg/L;		Volumetric (Imhoff cone), or gravimetric			2540 F [18 th , 19 th , 20 th , 21 st]	2540 F-97			
58. Residue, volatile mg/L;		Gravimetric, 550° C	160.4 ¹					1-3753-85 ²	
59. Rhodium- total ⁹ , mg/L;		Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA)							
		AA graphite furnace (GFAA)	265.2 ¹	7010	3111 B [18 th , 19 th , 21 st]	3111 B-99			
		Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
		Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A					
60. Ruthenium- Total ⁹ , mg/L;		Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA)							
		AA graphite furnace (GFAA)	267.2 ¹	7010	3111 B [18 th , 19 th , 21 st]	3111 B-99			
		Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
		Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A					
61. Selenium- Total ⁹ , mg/L;		Digestion ^{9, 11, 45} followed by: AA gaseous hydride		7741A ⁶	3114 B ¹⁰ [18 th , 19 th , 21 st]	3114 B-97 ¹⁰	D3859-98, 03 (A)	1-3667-85 ²	

Parameter Units	Analytical Technology ⁶⁸	EPA ^{44,62}	SW-846 ^{4,5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³		3113 B [18 th , 19 th , 21 st]	3113 B-99	D3859-98, 03 (B)	1-4668-98 ³⁹	
	Inductively coupled plasma-atomic emission spectrometry (ICP), or Inductively coupled plasma-mass spectrometry (ICP-MS)	200.7, Rev. 4.4 (1994) ¹³ 200.8, Rev. 5.4 (1994) ¹³	6010B, 6010C 6020A	3120 B [20 th , 21 st]	3120 B-99	D5673-03, 05		993.14 ¹
62. Silica- Dissolved ⁶⁶ , mg/L:	0.45 micron filtration followed by:							
	Colorimetric, Manual			4500-SiD [18 th , 19 th], 4500-SiO ₂ C [20 th , 21 st]	4500-SiO ₂ C-97	D859-94, 00, 05	1-1700-85 ²	
	Automated (Molybdsilicate)						1-2700-85 ²	
	Inductively coupled plasma-atomic emission spectrometry (ICP), or Inductively coupled plasma-mass spectrometry (ICP-MS)	200.7, Rev. 4.4 (1994) ¹³ 200.8, Rev. 5.4 (1994) ¹³	6010B, 6010C 6020A	3120 B [20 th , 21 st]	3120 B-99		1-4471-97 ⁶⁰	
63. Silver- Total ^{9, 18,40} , mg/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99		1-3720-85 ²	974.27 ¹
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99		1-4724-89 ⁶¹	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		1-4471-97 ²	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or Direct current plasma (DCP)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Digestion ^{9, 11, 45} followed by:							Note 43
64. Sodium- Total ⁹ , mg/L:	AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99		1-3735-85 ²	973.54 ¹

Parameter, Units	Analytical Technology ⁶⁵	EPA ⁴⁴ 62	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹¹	6010B, 6010C	3120 B [18 th , 19 th , 21 st]	3120 B-99		1-4471-97 ⁶⁶	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹²	6020A					
	Direct current plasma (DCP)							Note 43
	Flame photometric, or Ion chromatography			3500-Na D [18 th , 19 th], 3500-Na B [20 th , 21 st]	3500-Na B-97	D6919-03		
65. Specific conductance, micromhos/cm at 25° C:	Wheatstone bridge	120.1 ¹	9050A	2510 B [18 th , 19 th , 20 th , 21 st]	2510 B-97	D1125-95 (99), (05) (A)	1-2781-85 ²	973.40 ¹
66. Sulfate (as SO ₄ ²⁻), mg/L:	Automated colorimetric	375.2, Rev. 2.0 (1993)	9035					
	Gravimetric			4500-SO ₃ ²⁻ C or D [18 th , 19 th , 20 th , 21 st]			426C ¹⁹	925.54 ¹
	Turbidimetric					D516-90, 02		
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 th , 21 st]	4110 B-00	D4327-97, 03		993.30 ¹
	CIE/UV					D6508-00 (05)		D6508, Rev 2 ⁴⁴
67. Sulfide (as S), mg/L:	Titrimetric (iodine)			4500-S ²⁻ E [18 th], 4500-S ²⁻ F [19 th , 20 th , 21 st]	4500-S ²⁻ F-00		1-3840-85 ²	
	Colorimetric (methylene blue), or			4500-S ²⁻ D [18 th , 19 th , 20 th , 21 st]	4500-S ²⁻ D-00			
	Ion selective electrode			4500-S ²⁻ G [18 th , 19 th , 20 th , 21 st]	4500-S ²⁻ G-00	D4658-03		
68. Sulfite (as SO ₃), mg/L:	Titrimetric (iodine-iodate)			4500-SO ₃ ⁻ B [18 th , 19 th , 20 th , 21 st]	4500-SO ₃ ⁻ B-00			
	Colorimetric (methylene blue)			5540 C [18 th , 19 th , 20 th , 21 st]	5540 C-00	D2330-88, 02		
70. Temperature, °C:	Thermometric			2550 B [18 th , 19 th , 20 th , 21 st]	2550 B-00			Note 41

Parameter Units	Analytical Technology ^{6b}	EPA ^{44,62}	SW-846 ^{4,5}	Standard Methods [Editions] ⁶	Standard Methods- Online ⁷	ASTM ⁸	USGS	Other
71. Thallium- Total ⁹ , mg/L.	Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99			
	AA graphite furnace (GFAA)	279.2 (Issued 1978) ¹ , 200.9, Rev. 2.2 (1994) ¹³	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ³
72. Tin- Total ⁹ , mg/L.	Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA)		7000B	3111 B [18 th , 19 th , 21 st]	3111 B-99		1-3850-78 ¹⁶	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) ¹³	7010	3113 B [18 th , 19 th , 21 st]	3113 B-99			
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C					
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A					
73. Titanium- Total ⁹ , mg/L.	Digestion ^{9, 11, 45} followed by: AA direct aspiration (FLAA)			3111 D [18 th , 19 th , 21 st]	3111 D-99			
	AA graphite furnace (GFAA)	283.2	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or Direct current plasma (DCP)	200.8, Rev. 5.4 (1994) ¹³	6020A					Note 43

Parameter, Units	Analytical Technology ⁶⁸	EPA ^{44, 62}	SW-846 ^{4, 5}	Standard Methods [Editions] ⁶	Standard Methods-Online ⁷	ASTM ⁸	USGS	Other
74. Turbidity ⁶³ , NTU:	Nephelometric	180.1		2130 B [18 th , 19 th , 20 th , 21 st]	2130 B-01	D1889-94, 00	1-3860-85 ²	
75. Vanadium- Total ⁹ , mg/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 th , 19 th , 21 st]	3111 D-99			
	AA graphite furnace (GF AA)		7010			D3373-93, 03		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		14471-97 ⁶⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Direct current plasma (DCP), or							
	Colorimetric (Gallic acid)			3500-V D [18 th , 19 th], 3500-V B [20 th , 21 st]	3500-V B-97	D4190-94, 99, 03		Note 43
76. Zinc- Total ⁹ , mg/L:	Digestion ^{9, 11, 45} followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 th , 19 th , 21 st]	3111 B or C-99	D1691-95, 02 (A or B)	1-3900-85 ²	974.27 ¹ , P 37 ¹⁷
	AA graphite furnace (GF AA)	289.2 (Issued 1978) ¹	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) ¹³	6010B, 6010C	3120 B [20 th , 21 st]	3120 B-99		1-4471-97 ⁶⁰	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) ¹³	6020A			D5673-03, 05		993.14 ¹
	Direct current plasma (DCP)							
	Colorimetric (Dithizone), or			3500-Zn E [18 th , 19 th]		D4190-94, 99, 03		Note 43
	Colorimetric (Zincon)			3500-Zn F [18 th , 19 th], 3500-Zn B [20 th , 21 st]	3500-Zn B-97			Note 42

¹ "Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA-600/4-79-020 (NTIS PB 84-128677), Revised March 1983 and 1979 where applicable. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

² Fishman, M. J., et al. "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Denver, CO, Revised 1989, unless otherwise stated.

³ "Official Methods of Analysis of the Association of Official Analytical Chemists," Methods Manual, Sixteenth Edition, 4th Revision, 1998.

4. "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, September 1986 (Third edition), including July 1992 (Update I), September 1994 (Update II), August 1993 (Update IIA), January 1995 (Update IIB), December 1996 (Update III), April 1998 (Update IIIA), November 2004 (Update IIIB), February 2007 (Update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512-1800 (Publication Number: 955-001-00000-1). Also, available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.
5. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, Update IV includes methods 7000B, Flame Atomic Absorption Spectrophotometry and 7010, Graphite Furnace Atomic Absorption Spectrophotometry, general method descriptions.
6. "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, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.
7. "Standard Methods for the Examination of Water and Wastewater On-Line", Joint Editorial Board, American Public Health Association, American Water Works Association, Water Environment Federation, 2006.
8. "Standard Methods for the Examination of Water and Wastewater", American Society for Testing and Materials, 1994, 1996, 1999, 2007. Available from: the American Society for Testing and Materials service available at: <http://www.standardmethods.org>.
9. "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 19428-2959. Also available online at www.astm.org and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959.
10. For the determination of total metals (which are the equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-plateform graphite furnace atomic absorption determinations, a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA), a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R-94/111, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instructions and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (plateform furnace AA, STGFAA, ICP-AES, and ICP-MS) use EPA Method 200.2 or an approved alternate procedure (e.g. CEM microwave digestion, which may be used with certain analytes as indicated in Table B), the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.
11. Use the digestion given in the method.
12. "Test Methods for Evaluating Solid Waste", SW-846 method 3015A, United States EPA SW-846, 3rd Edition and updates. Footnote 4 lists the complete reference.
13. Copper sulfate may be used in place of mercuric sulfate.
14. "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), 5258 Port Royal Road, Springfield, Virginia 22161.
15. Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.
16. Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elnsford, NY 10523.
17. 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).
18. American National Standard on Photographic Processing Effluents, April 2, 1975. Available from ANSI, 25 West 43rd St., New York, NY 10036.
19. "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 (1981).
20. The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
21. Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD₅ is required can the permittee report data using a nitrification inhibitor.
22. OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.
23. Chemical Oxygen Demand, Method 8060, Hach Handbook of Water Analysis, 1979, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
24. The back titration method will be used to resolve controversy.
25. Orton Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Thermo Scientific, 81 Wymann Street, Waltham, MA 02454. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 mL 0.00281 N potassium iodate/100 mL solution, respectively.
26. The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976. Available on interlibrary loan.
27. "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.
28. Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
29. When using a method with block digestion, this treatment is not required.

- ²⁹ Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
- ³⁰ Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- ³¹ Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, Hach Chemical Company, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- ³² Wershaw, R.L., et al., "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- ³³ Nitrogen, Nitrite, Method 8507, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- ³⁴ Nitrate + nitrite determinations by ion chromatography must be analyzed within 48 hours.
- ³⁵ Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.
- ³⁶ The approved method is 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 spectrometric procedure. Available on interlibrary loan.
- ³⁷ R.F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421-426, 1970. Available in most public libraries.
- ³⁸ Back volumes of the Journal of Chromatography are available from: Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.
- ³⁹ 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 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 2 M Na₂S₂O₃ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
- ⁴⁰ The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition. Available on interlibrary loan.
- ⁴¹ For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH₄OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I₂ to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH > 7 with NH₄OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.
- ⁴² Stevens, H.H., Ficke, J.F. and Smoot, G.F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- ⁴³ Zinc, Zincon Method 8009, Hach Handbook of Water Analysis, 1979, Hach Company, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- ⁴⁴ "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991. Available from: Thermo Scientific, 81 Wyman Street, Waltham, MA 02454.
- ⁴⁵ Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals."
- ⁴⁶ Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", April 16, 1992, CEM Corporation, P.O. Box 200, Matthews, NC 28106-0200.
- ⁴⁷ When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.
- ⁴⁸ Only use n-hexane extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Method 1664A). Use of other extraction solvents (e.g., those in the 18th and 19th editions) is prohibited.
- ⁴⁹ Nitrogen, Total Kjeldahl, Method PA1-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- ⁵⁰ Nitrogen, Total Kjeldahl, Method PA1-DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- ⁵¹ Method 1664, Revision A "n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SCT-HEM; Non-polar Material) by Extraction and Gravimetry" EPA-821-R-98-002, February 1999. Available at NTIS, PB-121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.
- ⁵² The full text of Method 1650 is given in Appendix A, "Methods 1650 and 1653", of 40 CFR Part 430. Available from: The Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
- ⁵³ US EPA, 2001 Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" September 2002, Office of Water, U.S. Environmental Protection Agency (EPA-821-R-02-024). The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA-821-R-96-011) are recommended to preclude contamination at low-level, trace metal determinations. Available at NTIS, PB-121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.
- ⁵⁴ Quality control requirements for low level mercury are found in s. NR 106.145 (9) and (10), Wis. Adm. Code. Low-level mercury methods are performance based so some method modifications are allowable, provided quality control requirements are met. If an atomic absorption detector is substituted for atomic fluorescence detector, the appropriate method citation is 245.7.
- ⁵⁵ To eliminate the purge and trap step, the appropriate method citation is 245.7.
- ⁵⁶ Available Cyanide, Method OIA-1677, "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- ⁵⁷ "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method," Open File Report (OFR) 00-170.
- ⁵⁸ "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 93-449.

17 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 97-198.

18 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis," Open File Report (OFR) 92-146.

19 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace-Atomic Absorption Spectrometry," Open File Report (OFR) 98-639.

20 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry," Open File Report (OFR) 98-165.

21 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment," Open File Report (OFR) 93-125.

22 All EPA methods, excluding EPA Method 300.1, are published in "Methods for the Determination of Metals in Environmental Samples," Supplement 1, National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/R-94/111, May 1994; and "Methods for the Determination of Inorganic Substances in Environmental Samples," NERL-CI, EPA/600/R-93/100, August, 1993. EPA Method 300.1 is available from <http://www.epa.gov/safewater/methods/pdfs/met300.pdf>.

23 Styrene divinyl benzene beads (e.g. AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StablCalTM or equivalent) are acceptable substitutes for formazin.

24 Method D6508, Rev. 2, "Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte," available from Waters Corp, 34 Maple St., Milford, MA, 01757, Telephone: 508/482-2131, Fax: 508/482-3625.

25 Kelada-01, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821-B-01-009, Revision 1.2, August 2001, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001-108275]. The toll free telephone number is: 800-553-6847. Note: A 450-W UV lamp may be used in this method instead of the 550-W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.

26 QuikChem Method 10-204-00-1-X, "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis" is available from Hach Company, P.O. Box 389, Loveland, CO 80537.

27 When using sulfide removal test procedures described in Method 335.4, reconstitute particulate that is filtered with the sample prior to distillation.

28 Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation "followed by" analysis with a method, approved digestion and/or distillation are required prior to analysis.

29 Method 245.7, Rev. 2.0, "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry," February 2005, EPA-821-R-05-001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304.

30 The use of EDTA may decrease method sensitivity in some samples. Analysis may omit EDTA provided that all method specified quality control acceptance criteria are met.

31 Samples analyzed for available cyanide complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysis is further cautioned to limit the time between the addition of the ligand exchange reagents and sample analysis to no more than 30 minutes to preclude settling of materials in samples.

32 Oxygen, Dissolved, Luminescence, Hach Method 10360- Luminescence Measurement of Dissolved Oxygen (LDO®) in Water and Wastewater, Revision 1.1, January 2006, Hach Chemical Company, Loveland, CO 80537. Available from: Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.

Section 5: NIR 219.04 Table C is repeated and recreated to read:

Table C
List of Approved Analytical Methods for Non-Pesticide Organic Compounds

Parameter ¹	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
1. Acenaphthene	GC	610		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D			
	HP/LC	610	8310	6440 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
2. Acenaphthylene	GC	610		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
	GC/MS	625, 1625B	8270C, 8270D	6440 B [18 th , 19 th , 20 th , 21 st]		
3. Acrolein	HP/LC	610	8310			
	GC	603				
	GC/MS	625, 1624B				

Parameter ¹	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
4. Acrylonitrile	HPLC		8316			
	GC	603	8031			
5. Anthracene	GC/MS	624, 1624B				
	HPLC		8315A, 8316			Note 13, p.27
6. Benzene	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
	GC	602	8021B	6220 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
7. Benzidine	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	
	GC		8121			Note 6, p.1
8. Benzofluoranthracene	GC/MS	625 ⁸ , 1625B	8270C, 8270D			
	HPLC	605				
9. Benzotriptyrene	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
	GC	610				Note 13, p.27
10. Benzo(b)fluoranthene	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
11. Benzo(g,h,i)perylene	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
12. Benzo(k)fluoranthene	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
13. Benzyl chloride	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p. 130; Note 10, p. S102
14. Benzyl butyl phthalate	GC	606	8260B			
	GC/MS		8061A			
15. Bis(2-chloroethoxy) methane	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	611	8111			
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	611	8111			Note 13, p.27
17. Bis(2-ethylhexyl) phthalate	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	606				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
18. Bromodichloromethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
	GC	601	8020B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 B-97	
	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 C-97	
	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 B-97	
20. Bromomethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 C-97	
	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 B-97	
21. 4-Bromophenyl phenyl ether	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 C-97	
	GC	611	8111			Note 13, p.27
22. Carbon tetrachloride	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	
	GC/MS					

Parameter	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
23. 4-Chloro-3-methylphenol	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
24. Chlorobenzene	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.130
	GC	601, 602	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
25. Chloroethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 6, p.130
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
26. 2-Chloroethylvinyl ether	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
27. Chloroform	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
28. Chloromethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	612	8121	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
29. 3-Chloronaphthalene	GC/MS	625, 1625B	8270C, 8270D	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC	604	8041A	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
30. 2-Chlorophenol	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	611	8111	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
31. 4-Chlorophenyl phenyl ether	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	610	8310	6440 B [18 th , 19 th , 20 th]	6410 B-00	
32. Chrysene	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]	6410 B-00	
33. Dibenz(a,h)anthracene	GC/MS	610	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]	6410 B-00	
34. Dibromochloromethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	Note 13, p.27
	GC	601, 602	8260B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
35. 1,2-Dichlorobenzene	GC/MS	624, 1625B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	Note 13, p.27
	GC	601, 602	8260B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
36. 1,3-Dichlorobenzene	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	Note 13, p.27
	GC	601, 602	8260B, 8270C, 8270D	6230 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
37. 1,4-Dichlorobenzene	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	Note 13, p.27
	GC	601, 602	8260B, 8270C, 8270D	6230 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
38. 3,3-Dibromobenzidine	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	605	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
39. Dichlorodifluoromethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
40. 1,1-Dichloroethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
41. 1,2-Dichloroethane	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
42. 1,1-Dichloroethene	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	601	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
43. <i>trans</i> -1,2-Dichloroethene	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 13, p.27
	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
44. 2,4-Dichlorophenol	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	

Parameter ¹	Analytical Technology	EPA ^{2,11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
45. 1,2-Dichloropropane	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	
46. <i>cis</i> -1,3-Dichloropropene	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	
47. <i>trans</i> -1,3-Dichloropropene	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	
48. Diethyl phthalate	GC	606	8061A	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
49. 2,4-Dimethylphenol	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
50. Dimethyl phthalate	GC	606	8061A	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
51. Di- <i>n</i> -butyl phthalate	GC	606	8061A	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
52. Di- <i>n</i> -octyl phthalate	GC	606	8061A	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
53. 2,3-Dinitrophenol	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
54. 2,4-Dinitrophenol	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
55. 2,6-Dinitrophenol	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
56. 2,3-Dinitrotoluene	GC	609	8091	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
57. 2,4-Dinitrotoluene	GC	609	8091	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
58. 2,6-Dinitrotoluene	GC	609	8091	6410 B [18 th , 19 th , 20 th , 21 st]		Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
59. Epichlorohydrin	GC	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.130, Note 10, p. S102
	GC/MS	602	8260B			
60. Ethylbenzene	GC	602	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	
61. Fluoranthene	GC	610	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC/MS	625, 1625B	8310	6440 B [18 th , 19 th , 20 th]		
62. Fluorene	HPLC	610	8310	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	610	8310	6440 B [18 th , 19 th , 20 th]		
63. 1,2,3,4,6,7,8-Heptachlorodibenzofuran	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 th , 19 th , 20 th]		
64. 1,2,3,4,7,8,9-Heptachlorodibenzofuran	HRGC/LRMS	1613B ¹⁴	8280B			
	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
65. 1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin	HRGC/LRMS	1613B ¹⁴	8280B			
	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
	HRGC/LRMS		8280B			

Parameter ¹	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
66. Hexachlorobenzene	HRGC/HRMS GC	1613B ¹⁴ 612	8290A ¹⁴ 8081A, 8081B, 8121 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
67. Hexachlorobutadiene	GC/MS GC/MS	612 625, 1625B	8121 8260B, 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
68. Hexachlorocyclopentadiene	GC/MS	612 625, 1625B	8081A, 8081B, 8121 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
69. 1,2,3,4,7,8-Hexachlorodibenzofuran	HRGC/LRMS		8280B			
70. 1,2,3,6,7,8-Hexachlorodibenzofuran	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
71. 1,2,3,7,8,9-Hexachlorodibenzofuran	HRGC/LRMS		8280B			
72. 2,3,4,6,7,8-Hexachlorodibenzofuran	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
73. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			
74. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
75. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			Note 13, p.27
76. Hexachlorocycthane	HRGC/HRMS GC	1613B ¹⁴ 612	8290A ¹⁴ 8121	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
77. Indeno(1,2,3-cd)pyrene	GC/MS GC/MS	625, 1625B 610	8260B, 8270C, 8270D 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st] 6440 B [18 th , 19 th , 20 th]	6410 B-00	Note 13, p.27
78. Isophorone	HPLC GC	610 609	8310 8091			Note 13, p.27
79. Methylene chloride	GC/MS GC	625, 1625B 601	8270C, 8270D 8021B	6410 B [18 th , 19 th , 20 th , 21 st] 6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6410 B-00 6200 C-97	Note 6, p.130
80. 2-Methyl-4,6-dinitrophenol	GC/MS GC	624, 1624B 604	8260B 8041A	6210 B [18 th , 19 th], 6200 B [20 th , 21 st] 6420 B [18 th , 19 th , 20 th , 21 st]	6200 B-97 6420 B-00	Note 13, p.27
81. Naphthalene	GC/MS GC/MS	625, 1625B 610	8270C, 8270D 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00 6410 B-00	Note 13, p.27
82. Nitrobenzene	HPLC GC	610 609	8310 8091	6410 B [18 th , 19 th , 20 th , 21 st] 6440 B [18 th , 19 th , 20 th]	6410 B-00	Note 13, p.27
83. 2-Nitrophenol	GC/MS GC	625, 1625B 604	8270C, 8270D 8041A	6410 B [18 th , 19 th , 20 th , 21 st] 6420 B [18 th , 19 th , 20 th , 21 st]	6410 B-00 6420 B-00	Note 13, p.27
84. 4-Nitrophenol	GC/MS GC	625, 1625B 604	8270C, 8270D 8041A	6410 B [18 th , 19 th , 20 th , 21 st] 6420 B [18 th , 19 th , 20 th , 21 st]	6410 B-00 6420 B-00	Note 13, p.27

Parameter ¹	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
85. N-Nitrosodimethylamine	GC/MS GC	625, 1625B 607	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
86. N-Nitrosodi-n-propylamine	GC/MS	6255, 1625B		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
87. N-Nitrosodiphenylamine	GC/MS	6255, 1625B 607		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
88. Octachlorodibenzofuran	GC/MS	6255, 1625B		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
89. Octachlorodibenzo-p-dioxin	HRGC/LRMS HRGC/HRMS	1613B ¹⁴	8280B 8290A ¹⁴			
90. 2,2'-Oxybis(2-chloropropane)	HRGC/LRMS HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
91. PCB-1016 (Aroclor or congeners) ^{16, 17}	GC	611	8111			
92. PCB-1221 (Aroclor or congeners) ^{16, 17}	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.43, Note 12
93. PCB-1232 (Aroclor or congeners) ^{16, 17}	GC/MS HRGC/HRMS	608 625 1668A ¹⁸	8082 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.43, Note 12
94. PCB-1242 (Aroclor or congeners) ^{16, 17}	GC	608	8082			
95. PCB-1248 (Aroclor or congeners) ^{16, 17}	GC/MS HRGC/HRMS	625 1668A ¹⁸	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.43, Note 12
96. PCB-1254 (Aroclor or congeners) ^{16, 17}	GC	608	8082			
97. PCB-1260 (Aroclor or congeners) ^{16, 17}	GC/MS HRGC/HRMS	625 1668A ¹⁸	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.43, Note 12
98. 1,2,3,7,8-Pentachlorodibenzofuran	GC	608	8082			
99. 2,3,4,7,8-Pentachlorodibenzofuran	GC/MS HRGC/HRMS	625 1668A ¹⁸	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.43, Note 12
100. 1,2,3,7,8-Pentachlorodibenzo-p-dioxin	GC	608	8082			
	GC/MS HRGC/HRMS	625 1668A ¹⁸	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 6, p.43, Note 12
	HRGC/LRMS	1613B ¹⁴	8280B 8290A ¹⁴			
	HRGC/HRMS	1613B ¹⁴	8280B 8290A ¹⁴			
	HRGC/HRMS	1613B ¹⁴	8280B 8290A ¹⁴			
	HRGC/HRMS	1613B ¹⁴	8280B			

Parameter	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
101. Pentachlorophenol	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC	604	8041A	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D			
	GC	610		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
102. Phenanthrene	GC/MS	625, 1625B	8270C, 8270D	6440 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	HPLC	610	8310	6420 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	Note 13, p.27
	GC	604	8041A	6410 B [18 th , 19 th , 20 th , 21 st]		
103. Phenol	GC/MS	625, 1625B	8270C, 8270D			
	GC	610		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
104. Pyrene	GC/MS	625, 1625B	8270C, 8270D	6440 B [18 th , 19 th , 20 th]		
	HPLC	610	8310			
105. 2,3,7,8-Tetrachlorodibenzofuran	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴			
	HRGC/HRMS	601	8280B			
106. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	GC/MS	625 ⁹	8290A ¹⁴			
	HRGC/HRMS	1613B ¹⁴	8290A ¹⁴	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	Note 6, p.130
	HRGC/HRMS	601	8021B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
107. 1,1,2,2-Tetrachloroethane	GC	624, 1624B	8260B	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC/MS			6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
108. Tetrachlorocatechol	GC	1653 ¹⁵		6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	Note 6, p.130
	GC/MS	601	8021B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
109. Tetrachloroethene	GC	602	8260B	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC/MS	624, 1624B		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
110. Tetrachloroguaiacol	GC	1653 ¹⁵		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
	GC/MS	602	8021B	6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
111. 2,3,4,6-Tetrachlorophenol	GC	1653 ¹⁵		6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	Note 6, p.130, Note 13, p.27
	GC/MS	602	8260B			
112. Toluene	GC/MS	624, 1624B				
	GC	612	8021B			
113. 1,2,4-Trichlorobenzene	GC/MS	625, 1624B	8260B, 8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
	GC	612		6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
114. 3,4,5-Trichlorocatechol	GC/MS	1653 ¹⁵		6410 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC			6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
115. 3,4,6-Trichlorocatechol	GC/MS	1653 ¹⁵		6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
	GC	601	8021B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
116. 1,1,1-Trichloroethane	GC/MS	624, 1624B		6210 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
	GC	601	8021B	6200 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
117. 1,1,2-Trichloroethane	GC/MS	624, 1624B		6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
	GC	601	8021B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
118. Trichloroethene	GC/MS	624, 1624B		6230 B [18 th , 19 th , 20 th , 21 st]	6200 C-97	
	GC	601	8021B	6210 B [18 th , 19 th , 20 th , 21 st]	6200 B-97	
119. Trichloroethanemethane	GC/MS	624, 1624B		6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC	601	8021B	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
120. 3,4,5-Trichloroguaiacol	GC/MS	1653 ¹⁵		6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC			6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
121. 3,4,6-Trichloroguaiacol	GC/MS	1653 ¹⁵				

Parameter ¹	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods [Edition(s)] ⁴	Standard Methods Online ⁵	Other
122. 4,5,6-Trichloroquinol	GC			6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC/MS	1653 ¹⁵		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
123. 2,4,5-Trichlorophenol	GC			6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC/MS	1653 ¹⁵		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
124. 2,4,6-Trichlorophenol	GC	604	8041A	6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
125. Trichloroethylene	GC			6420 B [18 th , 19 th , 20 th , 21 st]	6420 B-00	
	GC/MS	1653 ¹⁵		6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	
	GC	601	8021B	6230 B [18 th , 19 th], 6200 C [20 th , 21 st]	6200 C-97	
126. Vinyl chloride	GC/MS	624, 1624B	8260B	6210 B [18 th , 19 th], 6200 B [20 th , 21 st]	6200 B-97	

1 All parameters are expressed in micrograms per liter (µg/L), except for Method 1613B in which the parameters are expressed in picograms per liter (pg/L).
2 The full text of Methods 601-613, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The full text of Method 1613B is incorporated by reference into this Part 136 and is available from the National Technical Information Services as stock number PB95-104774. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.
3 "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, September 1986 (third edition), including July 1992 (update I), September 1994 (update II), August 1993 (update IIA), January 1995 (update IIB), December 1996 (update III), April 1998 (update IIIA), November 2004 (update IIIB), February 2007 (update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512-1800 (Publication Number: 955-001-00000-1). Also, available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.
4 "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, 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.
5 "Standard Methods for the Examination of Water and Wastewater Online", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. Subscription service available at <http://www.standardmethods.org>.
6 "Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.
7 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 1624B.
8 Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.
9 5a 625, screening only.
10 "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 (1981).
11 Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601-603, 624, 625, 1624B, and 1625B (See Appendix A of this Part 136) in accordance with procedures each in Section 8.2 of each of these methods. Additionally, each laboratory, on an ongoing basis must spike and analyze 10% (5% for methods 624 and 625 and 100% for methods 1624B and 1625B) 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. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

12 "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk" 3M Corporation Revised 10/28/94.
13 USGS Method 0-3116-87 from "Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments," U.S. Geological Survey, Open File Report 93-125.
14 Analysis may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.
15 The full text of Method 1653 is given in Appendix A, "Methods 1650 and 1653", of 40 CFR Part 430. Available from: Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Also available on-line at <http://www.gpoaccess.gov/>.
16 EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non-detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration reported. It is recognized a number of congeners will co-elute with others, so there will not be 209 results to sum.
17 EPA Method 8082A shall be used for PCB-Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular

sample. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:

- 3620C – Florisil
- 3640A – Gel Permeation
- 3630C – Silica Gel
- 3611B – Alumina
- 3660B – Sulfur Clean Up
- 3665A – Sulfuric Acid Clean Up

18. Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS”, EPA-821-R-00-002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Section 6: NR 219.04 Table D is repealed and recreated to read:

Table D
List of Approved Analytical Methods for Pesticides¹ in Wastewater

Parameter	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods ⁴	Standard Methods Online ⁵	ASTM ⁶	Other
1. Aldrin	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00	D5812-96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]			Note 7, p. 83; Note 10, p. S68
	GC						Note 7, p. 94; Note 10, p. S16; Note 14
2. Aroclor	HPLC						Note 7, p. 83; Note 10, p. S68
3. Atrazine	LC/MS		8321A				Note 7, p. 83; Note 10, p. S68; Note 13
	GC						Note 7, p. 25; Note 10, p. S51
5. Atrazine	GC				6410 B-00		Note 7, p. 104; Note 10, p. S64; Note 14
6. Azinphos methyl	GC/MS		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		
7. Barban	HPLC			6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		
	GC/MS					D5812-96 (02)	Note 7, p. 7; Note 12
	LC/MS						
8. α -BHC	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00	D5812-96 (02)	Note 12
	GC/MS	625 ⁵	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]			
	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 12
9. β -BHC	GC	625 ⁵	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]			
	GC/MS	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625 ⁵	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]			
10. δ -BHC	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 7
	GC/MS	625 ⁵	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	D5812-96 (02)	Note 7, p. 94; Note 10, p. S60; Note 14
11. γ -BHC (lindane)	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]			
	GC/MS	625 ⁵	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]			
12. Captan	GC		8318		6410 B-00		
13. Carbaryl	HPLC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 8, p. 27; Note 10, p. S73
	GC/MS		8321A, 8325				
	LC/MS						
14. Carbofenthion	GC		8111B		6410 B-00		
	GC/MS		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	D5812-96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
15. Chlordane	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]			

Parameter	Analytical Technology	EPA ^{2, 11}	SW-846 ³	Standard Methods ⁴	Standard Methods Online ⁵	ASTM ⁶	Other
16. Chloropropham	GC/MS HPLC	625 ³	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 104; Note 10, p. S64; Note 14
17. 2,4-D	LC/MS GC		8321A	6640 B [18 th , 19 th , 20 th]			Note 7, p. 115; Note 8, p. 40
18. 4,4'-DDE	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]		D5812-96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
19. 4,4'-DDE	GC/MS GC	625 608	8270C, 8270D 8081A, 8081B	6410 B [18 th , 19 th , 20 th , 21 st] 6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
20. 4,4'-DDT	GC/MS GC	625 608	8270C, 8270D 8081A, 8081B	6410 B [18 th , 19 th , 20 th , 21 st] 6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
21. DDT	GC/MS GC	625	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
22. DDT	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 10, p. S51
23. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
24. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
25. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
26. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
27. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
28. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
29. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
30. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
31. Dieldrin	GC/MS GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
32. Endosulfan I	GC/MS GC	608 625 ⁹	8081A, 8081B 8270C, 8270D	6630 B and C [18 th , 19 th , 20 th] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
33. Endosulfan II	GC/MS GC	608 625	8081A, 8081B 8270C, 8270D	6630 B and C [18 th , 19 th , 20 th , 21 st] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
34. Endosulfan sulfate	GC/MS GC	608 625	8081A, 8081B 8270C, 8270D	6630 B and C [18 th , 19 th , 20 th , 21 st] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 12
35. Endosulfan	GC/MS GC	608 625 ⁹	8081A, 8081B 8270C, 8270D	6630 B and C [18 th , 19 th , 20 th , 21 st] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
36. Endosulfan	GC/MS GC	608 625	8081A, 8081B 8270C, 8270D	6630 B and C [18 th , 19 th , 20 th , 21 st] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 12
37. Endosulfan	GC/MS GC	625	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 8, p. 27; Note 10, p. S73
38. Endosulfan	GC/MS HPLC	625	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 104; Note 10, p. S64; Note 14
39. Endosulfan	GC/MS HPLC	608 625 ⁹	8081A, 8081B 8270C, 8270D	6630 B and C [18 th , 19 th , 20 th] 6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 104; Note 10, p. S64; Note 14
40. Endosulfan	GC/MS GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 12
41. Endosulfan	GC/MS GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 7; Note 8, p. 27; Note 10, p. S73;

Parameter	Analytical Technology	EPA ¹¹	SW-846 ¹	Standard Methods ⁴	Standard Methods Online ⁵	ASTM ⁶	Other
	GC/MS	625 ⁴	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 12
42. Isodrin	GC		8081A, 8081B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 104; Note 10, p. S64; Note 14
43. Lururon	HPLC		8321A, 8325				Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
	LC/MS			6630 C [18 th , 19 th , 20 th]			
44. Malathion	GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 94; Note 10, p. S60; Note 14
	GC/MS		8318				
45. Methiocarb	HPLC		8321A, 8325			D5812-96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	LC/MS			6630 B and C [18 th , 19 th , 20 th]			
46. Methoxychlor	GC/MS		8081A, 8081B	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 94; Note 10, p. S60; Note 14
	GC/MS		8270C, 8270D				
47. Mefenacarbate	HPLC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 7; Note 8, p. 27
	GC/MS		8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]			
48. Mirex	GC		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		Note 7, p. 104; Note 10, p. S64; Note 14
	GC/MS						
49. Monuron	HPLC		8321A, 8325				Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS						Note 7, p. 104; Note 10, p. S64; Note 14
50. Monuron-1CA	HPLC						
51. Neburon	HPLC						
	LC/MS						
52. Parathion methyl	GC		8321A	6630 C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 25; Note 8, p. 27
	GC/MS		8141A, 8141B	6410 B [18 th , 19 th , 20 th , 21 st]			
	GC/MS		8270C, 8270D				
53. Parathion ethyl	GC		8321A	6630 C [18 th , 19 th , 20 th]	6410 B-00		Note 7, p. 25; Note 8, p. 27
	GC/MS		8141A, 8141B	6410 B [18 th , 19 th , 20 th , 21 st]			
	GC/MS		8270C, 8270D	6630 B and C [18 th , 19 th , 20 th]			
54. PCNB	GC		8081A, 8081B	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00	D5812-96 (02)	Note 8, p. 27
	GC/MS		8270C, 8270D				Note 7, p. 83; Note 10, p. S68; Note 13
55. Perthane	GC		8081A, 8081B				Note 7, p. 83; Note 10, p. S68; Note 13
56. Prometon	GC						Note 7, p. 83; Note 10, p. S68; Note 13
57. Prometryn	GC						Note 7, p. 83; Note 10, p. S68; Note 13
58. Propazine	GC						Note 7, p. 104; Note 10, p. S64; Note 14
59. Propitiam	HPLC						
	LC/MS		8321A				Note 7, p. 94; Note 10, p. S60; Note 14
60. Propoxir	HPLC		8318				
	LC/MS		8321A				Note 7, p. 83; Note 10, p. S68; Note 14
61. Sebumeton	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
62. Sidoron	HPLC						
	LC/MS		8321A, 8325				Note 7, p. 83; Note 10, p. S68; Note 13
63. Simazine	GC		8141A, 8141B				Note 7, p. 7
64. Strobane	GC		8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]			Note 7, p. 104; Note 10, p. S64; Note 14
65. Swept	HPLC						Note 7, p. 115; Note 8, p. 40
66. 2,4,5-T	GC		8151A	6640 B [18 th , 19 th , 20 th]			
	GC		8321A				Note 7, p. 115; Note 8, p. 40
67. 2,4,5-TP (Silvex)	LC/MS		8151A	6640 B [18 th , 19 th , 20 th]			
	GC		8321A				Note 7, p. 83; Note 10, p. S68
68. Terbutylazine	GC						

Parameter	Analytical Technology	EPA ^{1, 11}	SW-846 ³	Standard Methods ⁴	Standard Methods Online ⁵	ASTM ⁶	Other
69. Toxaphene	GC	608	8081A, 8081B	6630 B and C [18 th , 19 th , 20 th]		D5812-96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		
70. Trifluralin	GC		8081A, 8081B	6630 B [18 th , 19 th , 20 th]			Note 7, p. 7; Note 13
	GC/MS		8270C, 8270D	6410 B [18 th , 19 th , 20 th , 21 st]	6410 B-00		

¹ Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.
² The full text of Methods 608 and 625 are given at Appendix A. "Test Procedures for Analysis of Organic Pollutants," 40 CFR, Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B. "Definition and Procedure for the Determination of the Method Detection Limit," 40 CFR, Part 136.
³ "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 (third edition), including July 1992 (update I), September 1994 (update II), August 1993 (update IIA), January 1995 (update IIB), December 1996 (update III), April 1998 (update IIIA), November 2004 (update IIIB), February 2007 (update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512-1800 (Publication Number: 955-001-00000-1). Also, available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

⁴ "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, 21st Edition (2001), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005
⁵ "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, 2006. On-line subscription service available at <http://www.standardmethods.org>
⁶ "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 2007. Available from: the American Society for Testing and Materials, 100 Burr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. Also available online at www.astm.org.

⁷ "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 (HPLC) methods.
⁸ "Methods for Analysis of Organic Substances in Water and Fluvial Sediments", Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).
⁹ The method may be extended to include a-BHC, g-BHC, endosulfan I, and endrin. However, when they are known to exist, Method 608 is the preferred method.
¹⁰ "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 (1981).

¹¹ 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, 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. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

¹² "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk", 3M Corporation, Revised 10/28/94.
¹³ USGS Method 0-3106-93 from "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors" U.S. Geological Survey Open File Report 94-37.
¹⁴ 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.

Section 7: NR 219.04 Table E is repealed and recreated to read:

Table E
List of Approved Radiological Analytical Methods for Wastewater

Parameter and Units	Analytical Technology	EPA ¹	Standard Methods ²	Standard Methods Online ³	ASTM ⁴	USGS ⁵
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.0	7110 B	7110 B-00	D1943-05	pp. 75 and 78 ⁶
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	7110 B-00	D1943-05	p. 79
3. Beta-Total, pCi per liter.	Proportional counter	900.0	7110 B	7110 B-00	D1890-05	pp. 75 and 78 ⁶
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	7110 B-00	D1890-05	p. 79
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500-Ra B	7500-Ra B-01	D2460-05	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500-Ra C	7500-Ra C-01	D3454-05	p. 81

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

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, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

³ "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, 2006. On-line subscription service available at <http://www.standardmethods.org>.

⁴ "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. Also available online at www.astm.org.

⁵ Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

⁶ The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

Section 8: NR 219.04 Table EM is repealed and recreated to read:

Table EM
List of Approved Analytical Methods for Sludge

Parameter	Analytical Technology	Sample Preparation		Method			Other
		SW-846 ¹	EPA ⁴	SW-846 ¹	EPA ^{2,3}	Standard Methods ^{8,9}	
Metals							
Arsenic	Gaseous Hydride ⁵	7061A		7061A			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 th , 19 th , 21 st], 3113 B-99	
		3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A		6020A	200.8		
Beryllium	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A					
	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 D [18 th , 19 th , 21 st], 3111 D-99	
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 th , 19 th , 21 st], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
Cadmium	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
	Flame Atomic Absorption	3050B, 3051A	200.2	7000B			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 th , 19 th , 21 st], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	

Parameter	Analytical Technology	Sample Preparation		Method			
		SW-846 ¹	EPA ⁴	SW-846 ¹	EPA ^{2,3}	Standard Methods ^{8,9}	Other
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Chromium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B [18 th , 19 th , 21 st], 3111 B-99	
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 th , 19 th , 21 st], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Copper	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18 th , 19 th , 21 st], 3111 B-99 or C-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Lead	Flame Atomic Absorption	3050B, 3051A	200.2	7000B			
	Graphite Furnace ⁶	3050B, 3051A	200.2	7010	200.9	3113 B [18 th , 19 th , 21 st], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Mercury	Cold Vapor Atomic Absorption	7471A, 7471B		7471A, 7471B			
	Cold vapor atomic fluorescence spectrometry	7474					
Molybdenum	Graphite Furnace ⁶	3050B, 3051A	200.2	7010	200.9	3113 B [18 th , 19 th , 21 st], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Nickel	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18 th , 19 th , 21 st], 3111 B-99 or C-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Selenium	Gaseous Hydride ⁵	7741A		7741A			
	Graphite Furnace	3050B, 3051A	200.2	7010		3113 B [18 th , 19 th , 21 st], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Zinc	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18 th , 19 th , 21 st], 3111 B-99 or C-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 th , 21 st], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Organics							

Parameter	Analytical Technology	Sample Preparation		Method			
		SW-846 ¹	EPA ⁴	SW-846 ¹	EPA ^{2,3}	Standard Methods ^{8,9}	Other
Dioxins and Furans	Gas Chromatography/Mass Spectrometry	8290A ¹¹	1613B ¹¹	8290A	1613B		
PCB (Aroclor or Congeners)	Gas Chromatography	3540B, 3540C, 3545A		8082, 8082A ¹²			
PCB (Congeners)	Gas Chromatography/Mass Spectrometry	1668A ^{13, 14, 15}			1668A ^{13, 14, 15}		
Biological							
Enteric Viruses	Centrifuge Concentration						D 4994-89 (02) ⁷ , or Appendix H ¹⁰
Fecal Coliform	Most Probable Number Membrane Filter					9221 E [18 th , 19 th , 20 th , 21 st], 9221 E-99, 9222 D, 9222 D-97	Appendix F ¹⁰
Helminth ova	Density Gradient Flotation						Note 9 or Appendix I ⁹
Specific Oxygen Uptake Rate	Respirometer					2710 B [18 th , 19 th , 20 th , 21 st], 2710 B-04	Appendix D.2. ¹⁰
Salmonella	Most Probable Number Selective Media Culture						9260 D.1 ⁸ or Appendix G ¹⁰
Physical							
Solids	Gravimetric					2540 G [18 th , 19 th , 20 th , 21 st], 2540 G-97	
Percent Volatiles Solids Reduction	Calculation						Appendix D.1. and 3 ¹⁰

¹ "Test Methods for Evaluating Solid Waste", Physical/Chemical Methods, SW-846, Environmental Protection Agency, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (Third edition), including July 1992 (Update I), September 1994 (Update II), August 1993 (Update IIA), January 1995 (Update IIB), December 1996 (Update III), April 1998 (Update IIIA), November 2004 (Update IIIB), February 2007 (Update IV) updates. Available from: The Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250-7954. Available online at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

² If an alternative digestion procedure is specified in the analytical method, the digestion in this table shall be used. In all cases, consult the analytical method for special requirements and cautions. SW-846 method 3051A is an acceptable alternate digestion procedure to SW-846 method 3050B.

³ "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), 5258 Port Royal Road, Springfield, Virginia 22161.

⁴ "Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements", Method 200.2, Revision 2.8, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, 1994. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.

⁵ High levels of chromium, copper, mercury, silver, cobalt, or molybdenum may interfere with the analysis. Consult Method 3114, of "Standard Methods for the Examination of Water and Wastewater", 18th, 19th, 20th, or 21st edition, for more information.

⁶ Concentrations of lead in municipal sludge may exceed the working range of graphite furnace.

⁷ 1993 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.

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, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

⁹ "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On-line subscription service available at <http://www.standardmethods.org>.

¹⁰ "Occurrence of Pathogens in Distribution and Marketing Municipal Sludges", EPA 600/1-87-014, Environmental Protection Agency, 1987. Available from: the National Technical Information Service, order # PB 88-154273/AS, 5285 Port Royal Road, Springfield, Virginia 22161.

¹¹ "Environmental Regulations and Technology - Control of Pathogens and Vectors Attraction in Sewage Sludge", EPA-625/R-92/013, Revised October 1999, Environmental Protection Agency, Cincinnati, OH, 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

¹² Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

¹³ EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non-detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration by dry weight reported. It is recognized that a number of the congeners will co-elute with others, so there will not be 209 results to sum.

¹⁴ EPA Method 8082A shall be used for PCB-Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular sample. For either type of analysis, the sample shall be extracted using Soxhlet extraction Method 3540C or Pressurized Fluid Extraction Method 3545A. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and achieve as close to a limit of detection of 0.11 mg/kg as possible. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and to achieve as close to a limit of detection of 0.003 mg/kg as possible for each congener. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:

- 3620C – Florisil
- 3640A – Gel Permeation
- 3630C – Silica Gel
- 3611B – Alumina
- 3660B – Sulfur Clean Up
- 3665A – Sulfuric Acid Clean Up.

¹⁵ "Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS", EPA-821-R-00-002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Section 9: Table F is repealed and recreated to read:

Table F
Required Containers, Preservation Techniques, and Holding Time for Wastewater

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum Holding Time ⁴
Table A— Bacterial Tests:			
1-5. Coliform, total, fecal and <i>E. coli</i>	PA, G	Cool, <10°C, 0.0008% Na ₂ SO ₃ ⁵	6 hours
6. Fecal streptococci	PA, G	Cool, <10°C, 0.0008% Na ₂ SO ₃ ⁵	6 hours
7. Enterococci	PA, G	Cool, <10°C, 0.0008% Na ₂ SO ₃ ⁵	6 hours
Table A: Protozoan Tests:			
8. Cryptosporidium	LDPE, field filtration	0-8°C	96 hours ²¹
9. Giardia	LDPE, field filtration	0-8°C	96 hours ²¹
Table A: Aquatic Toxicity Tests			
10-11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6°C ¹⁶	36 hours
Table B— Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6°C ¹⁸	14 days
2. Alkalinity	P, FP, G	Cool, ≤6°C ¹⁸	14 days
4. Ammonia	P, FP, G	Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6°C ¹⁸	48 hours
11. Bromide	P, FP, G	None required	28 days
14. Carbonaceous biochemical oxygen demand	P, FP, G	Cool, ≤6°C ¹⁸	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6°C ¹⁸	48 hours
23-24. Cyanide, total or available (CATC)	P, FP, G	Cool, ≤6°C ¹⁸ , NaOH to pH>12 ⁶ , reducing agent ⁵	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 44. Kjeldahl and organic N	P, FP, G	Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2	28 days
38. Nitrate	P, FP, G	Cool, ≤6°C ¹⁸	48 hours
39. Nitrate + nitrite	P, FP, G	Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6°C ¹⁸	48 hours
41. Oil and grease	G	Cool, ≤6°C ¹⁸ , HCl or H ₂ SO ₄ to pH<2	28 days
42. Organic carbon	P, FP, G	Cool, ≤6°C ¹⁸ , HCl, H ₂ SO ₄ or H ₃ PO ₄ to pH<2	28 days

Parameter number/name	Container ¹	Preservation ^{2, 4}	Maximum Holding Time ⁴
45. Orthophosphate	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	Analyze within 48 hours; filter, if needed, within 15 minutes ²²
47. Oxygen, dissolved (Probe or Luminescence)	G, Bottle and top	None required	Analyze within 15 minutes
47. Oxygen, dissolved (Winkler)	G, Bottle and top	Fix on site and store in dark	8 hours
49. Phenols	G	Cool, $\leq 6^{\circ}\text{C}^{18}$, H_2SO_4 to pH<2	28 days
50. Phosphorus (elemental)	G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours
51. Phosphorus, total	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$, H_2SO_4 to pH<2	28 days
54. Residue, total	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days
55. Residue, filterable	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days
56. Residue, nonfilterable (TSS)	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours
57. Residue, settleable	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days
58. Residue, volatile	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	28 days
62. Silica	P or Quartz	Cool, $\leq 6^{\circ}\text{C}^{18}$	28 days
65. Specific conductance	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	28 days
66. Sulfate	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	28 days
67. Sulfide	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$, add zinc acetate plus sodium hydroxide to pH>9	7 days
68. Sulfite	P, FP, G	None required	Analyze within 15 minutes
69. Surfactants	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours
70. Temperature	P, FP, G	None required	Analyze within 15 minutes
74. Turbidity	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours
Table B—Metals: ⁹			
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 46, 48, 52, 53, 59-61, 63, 64, 71-73, 75, 76. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO_3 to pH<2, or at least 24 hours prior to analysis ¹⁹	6 months
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months
18. Chromium IV	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$, pH = 9.3 - 9.7 ²⁰	28 days
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH<2	28 days
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹⁷	5 mL/L 12N HCl or 5 mL/L BrCl^{17}	90 days ¹⁷
Table C—Organic Tests ⁸			
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5	14 days ¹⁰
7, 38. Benzidines ^{11, 12}	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5	7 days until extraction ¹³
29, 35-37, 66-68, 76, 113. Chlorinated hydrocarbons ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 90. Haloethers	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5	7 days until extraction, 40 days after extraction
56-58, 78, 82. Nitroaromatics and Isophorone ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, store in dark, 0.0008% Na_2SO_3^5	7 days until extraction, 40 days after extraction
85-87. Nitrosamines ^{11, 14}	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, store in dark, 0.0008% Na_2SO_3^5	7 days until extraction, 40 days after extraction
91-97. PCBs ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$	1 year until extraction, 1 year after extraction
63-65, 69-75, 88, 89, 98-100, 105, 106. PCDDs/PCDFs ¹¹			
Aqueous Samples: Field and Laboratory Preservation	G	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5 , pH<9	1 year
Solids and Mixed Phase Samples: Field Preservation	G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days
Tissue Samples: Field Preservation	G	Cool, $\leq 6^{\circ}\text{C}^{18}$	24 hours
Solids, Mixed-Phase, and Tissue Samples: Laboratory Preservation	G	Freeze, $\leq -10^{\circ}\text{C}$	1 year
23, 30, 44, 49, 53-55, 80, 83, 84, 101, 103, 108, 110, 111, 114, 115, 120-125. Phenols ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5	7 days until extraction, 40 days after extraction
14, 17, 48, 50-52. Phthalate esters ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5	7 days until extraction, 40 days after extraction ¹⁵
1, 2, 5, 8-12, 32, 33, 61, 62, 77, 81, 102, 104. Polynuclear aromatic hydrocarbons ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, store in dark, 0.0008% Na_2SO_3^5	7 days until extraction, 40 days after extraction
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 59, 79, 107, 109, 113, 116-119, 126. Purgeable halocarbons.	G, FP-lined septum	Cool, $\leq 6^{\circ}\text{C}^{18}$, 0.0008% Na_2SO_3^5	14 days
6, 60, 112. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, $\leq 6^{\circ}\text{C}^{18}$, HCl to pH<2, 0.0008% Na_2SO_3^5	14 days ⁹
Table D. Pesticides Tests			
1-70. Pesticides ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}^{18}$, pH 5-9 ¹⁵	7 days until extraction, 40 days after extraction
Table E—Radiological Tests			
1-5. Alpha, beta and radium	P, FP, G	HNO_3 to pH<2	6 months

¹ "P" is polyethylene; "FP" is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table F; "G" is glass; "PA" is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); "LDPE" is low density polyethylene.

² Except where noted in this Table F each grab sample shall be preserved within 15 minutes of collection. For a composite sample collected with an automated sampler, refrigerate the sample at $\leq 6^{\circ}\text{C}$ during collection unless specified elsewhere in this table or in the method(s). For a composite sample to be split into separate aliquots for preservation and analysis, maintain the sample at $\leq 6^{\circ}\text{C}$, unless specified elsewhere in this table or in the method(s) until collection, splitting and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of the sample. Preserve (e.g., addition of acid, base or other chemical) the grab sample, composite sample, or aliquot split from the composite sample within 15 minutes of collection. 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. 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.

³ When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, 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 II, 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 the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator (s. NR 219.05). A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability.

⁵ Add a reducing agent only in the presence of residual chlorine.

⁶ Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to >12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to >12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH >12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary. **Sulfur:** To remove elemental sulfur (S₈), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to >12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in μg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(1) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4-L collapsible container (e.g., Cubitainer™). Acidify with concentrated hydrochloric acid to pH <2 . Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to >12 , refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide ($>10\%$). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH <2 . Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to >12 , refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide ($>10\%$). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in μg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to >12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with

concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in g or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand exchange reagent to offset any excess of cadmium chloride.

(2) Sulfite, thiosulfate, or thiocyanate: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA-1677) to preclude cyanide loss or positive interference.

(3) Aldehyde: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(4) Carbonate: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥ 12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500-CN.B.3.d).

(5) Chlorine, hypochlorite, or other oxidant: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

⁷ For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler, filter the sample within 15 minutes after completion of collection and before adding preservatives.

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

⁹ If the sample is not adjusted to $\text{pH} < 2$, then the samples 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 three days of sampling.

¹¹ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to $\leq 6^\circ\text{C}$, 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 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding 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 30 days at $< 0^\circ\text{C}$.

¹⁴ For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ 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% $\text{Na}_2\text{S}_2\text{O}_3$.

¹⁶ Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that $\leq 6^\circ\text{C}$ has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

¹⁷ Samples collected for the determination of trace level mercury ($< 100 \text{ ng/L}$) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

¹⁸ Aqueous samples must be preserved at $\leq 6^\circ\text{C}$, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. The specification of " $\leq 6^\circ\text{C}$ " is used in place of the " 4°C " and " $< 4^\circ\text{C}$ " sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6°C may not be used to meet the $\leq 6^\circ\text{C}$ requirement. The preservation temperature does not apply to samples that must be analyzed within 15 minutes.

¹⁹ An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

²⁰ To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

²¹ Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

²² Filtration is only required when reporting dissolved orthophosphate, dissolved hydrolyzable phosphorus or dissolved organic phosphorus as described in EPA Method 365.1 (1993). Filtration must be completed within 15 minutes of collection using a $0.45\mu\text{m}$ filter, sample shall be maintained at 6°C and analyzed within 48 hours.

SECTION 10. Effective date. This rule shall take effect the first day of the month following publication in the Wisconsin administrative register as provided in s. 227.22(2)(intro.), Stats.

SECTION 11. Board adoption. This rule was approved and adopted by the State of Wisconsin Natural Resources Board on December 10, 2008.

Dated at Madison, Wisconsin _____.

STATE OF WISCONSIN
DEPARTMENT OF NATURAL RESOURCES

By _____
Matthew Frank, Secretary

(SEAL)