**State of Wisconsin**
**DEPARTMENT OF NATURAL RESOURCES**
**101 S. Webster Street**
**Box 7921**
**Madison WI 53707-7921**

**Scott Walker, Governor**
**Cathy Stepp, Secretary**
Telephone 608-266-2621
Toll Free 1-888-936-7463
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**Notice Soliciting Comments Regarding an Economic Impact Analysis**

**Subject:** Revisions to ch. NR 219, analytical test methods and procedures for wastewater analysis
**Notice Date:** November 15, 2013
**Comment Period:** November 15, 2013 to November 29, 2013

The Department of Natural Resources is in the process of preparing an economic impact analysis (EIA) for proposed rules relating to ch. NR 219, Wastewater analytical test methods and procedures, Board Order SS-14-12. A preliminary draft of the EIA and a draft of the rule order are available for download as a clickable link by going to the following site: http://dnr.wi.gov and searching for the keywords "Administrative Rules" or at http://adminrules.wisconsin.gov .

If you are not able to access or download the information, please send an email to the following address: Richard.Mealy@wisconsin.gov

Pursuant to s. 227.137, Wis. Stats., the department is required to solicit comments on the economic impact of proposed rule SS-14-12 and, if requested, to coordinate with local governments in the preparation of the EIA. Notice is hereby given that the Department of Natural Resources will accept written comments on the EIA until November 15, 2013. Comments may be submitted electronically to: Richard.Mealy@wisconsin.gov or may be mailed to: Richard Mealy- SS/7, Wisconsin Dept. of Natural Resources, P.O. Box 7921, Madison, WI 53707.

Any local governmental unit that is affected by the rule may also request to coordinate with the department on preparation of the EIA. If a local governmental unit wishes to coordinate with the department on preparation of the EIA, the governmental unit must notify the department of its request to coordinate at the time comments on the EIA are submitted. The department will then contact all local governmental units requesting an opportunity to coordinate and incorporate their comments into the EIA to the extent feasible.

Pursuant to Executive Order # 50 (2011) and s. 227.137, Wis. Stats., the department must include the information listed below in an EIA. To review all of the information that must be included in an EIA, you may refer to the Executive Order and statutory provisions. When submitting comments, please provide specific information in these areas and include any supporting economic data, studies or reports. Please do NOT submit comments on revisions to the rule language at this time. The department is soliciting information on the following from you and others:

> Would you, your business, your association, or your local unit of government be affected in a material economic way by the implementation of these changes to the approved methodologies for the analysis of wastewater?

Additionally, the department is seeking comments on the following from people who indicate they will be affected economically:

1. Any implementation or compliance costs that are reasonably expected to be incurred.
2. Actual quantifiable benefits of the proposed rule.
3. Whether the proposed rule would adversely affect in a material way the economy, a sector of the economy, productivity, jobs, or the overall economic competitiveness of the state.

4. Economic impacts of specific alternatives to the proposed rule.
5. Whether the proposed rule will have an economic impact (savings or increased costs) on public utilities or their ratepayers.

If you are a small business as defined in s. 227.114(1), Wis. Stats., please identify yourself as a small business in your comments. Small business means a business entity, including its affiliates, which is independently owned and operated and not dominant in its field, and which employs 25 or fewer full-time employees or which has gross annual sales of less than $5,000,000.

Please refer to s. 227.19(3)(e)3. and 4. for further information when you are preparing your comments as a small business. Following the public comment period for the EIA, a revised "Fiscal Analysis and Economic Impact Analysis" will be prepared containing relevant information that the department receives. Once the EIA process is completed, the department will submit the rule package and economic impact analysis to the Wisconsin Legislative Council under s. 227.15, Wis. Stats., and hearings on the proposed rule will be held by the department after proper notice in accordance with ss. 227.17 and 227.18, Wis. Stats. If the EIA indicates that the proposed rule is reasonably expected to have a total impact of $20,000,000 in implementation and compliance costs, the department shall submit the rule to the Department of Administration in accordance with s. 227.137(6), Wis. Stats.

NOTE: Chapter 227 of the statutes may be found at the following web site: https://docs.legis.wisconsin.gov/statutes/statutes/227.pdf.

Further information on the process for enacting rules is contained in Executive Order #50 signed by Governor Walker on November 2, 2011 (available at http://docs.legis.wisconsin.gov/code/executive_orders/2011_scott_walker/2011-50).

STATE OF WISCONSIN
DEPARTMENT OF ADMINISTRATION
DOA-2049 (R03/2012)

DIVISION OF EXECUTIVE BUDGET AND FINANCE
101 EAST WILSON STREET, 10TH FLOOR
P.O. BOX 7864
MADISON, WI 53707-7864
FAX: (608) 267-0372

# ADMINISTRATIVE RULES
# Fiscal Estimate & Economic Impact Analysis

1. Type of Estimate and Analysis
☒ Original ☐ Updated ☐ Corrected

2. Administrative Rule Chapter, Title and Number
NR 219

3. Subject
Analytical test procedures and approved methods.

4. Fund Sources Affected
☐ GPR ☐ FED ☐ PRO ☐ PRS ☐ SEG ☐ SEG-S

5. Chapter 20, Stats. Appropriations Affected
none

6. Fiscal Effect of Implementing the Rule
☒ No Fiscal Effect ☐ Increase Existing Revenues ☐ Increase Costs
☐ Indeterminate ☐ Decrease Existing Revenues ☐ Could Absorb Within Agency's Budget
☐ Decrease Cost

7. The Rule Will Impact the Following (Check All That Apply)
☐ State's Economy ☒ Specific Businesses/Sectors
☒ Local Government Units ☐ Public Utility Rate Payers
☐ Small Businesses **(if checked, complete Attachment A)**

8. Would Implementation and Compliance Costs Be Greater Than $20 million?
☐ Yes ☒ No

9. Policy Problem Addressed by the Rule
Current wastewater analysis methods being used are not in compliance with EPA requirements. The EPA is requiring the WDNR to remove the SW 846 methods for wastewater analysis from the rule. SW 846 methods will still be included in NR 219 and allowed to be used for the analysis of sludge (currently Table EM). In addition, we will be adding methods the EPA has promulgated in the Method Update Rule (MUR) dated May 18, 2012.

10. Summary of the businesses, business sectors, associations representing business, local governmental units, and individuals that may be affected by the proposed rule that were contacted for comments.
Laboratories using SW 846 methods could potentially be affected. These include small businesses, businesses, and municipal wastewater treatment facilities (WWTF). In our preliminary research we conducted a survey of 57 labs that could potentially be using SW-846 methods.

We received 19 responses. Sixty-seven percent (thirteen respondents) anticipated no economic impact. Thirty-three percent (six respondents) anticipated some, but minimal economic impact.

11. Identify the local governmental units that participated in the development of this EIA.
See above.

12. Summary of Rule's Economic and Fiscal Impact on Specific Businesses, Business Sectors, Public Utility Rate Payers, Local Governmental Units and the State's Economy as a Whole (Include Implementation and Compliance Costs Expected to be Incurred)
We expect the economic and fiscal impact of this rule revision to be minimal. Those who responded to our survey reported costs ranging from $50 to $25,000. One lab did not provide a cost estimate. Four of the six respondents estimated costs less than $4,500.

To estimate the economic impact statewide, we assume (based on responses) that 30 percent of state labs will need to revise their methods.

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# ADMINISTRATIVE RULES
## Fiscal Estimate & Economic Impact Analysis

The majority of the costs come from staff time spent revising SOPs and validating the new methods. Alternative methods are available online for free through the EPA. Methods are similar enough that laboratory equipment and supplies will likely be comparable in cost. Costs to laboratories will primarily be incurred from time spent validating new methods and revising SOPs.

A survey was sent out to 57 labs known or believed to perform wastewater testing using the methodfs wscheduled to be removed., There were 19 respondents (33% response rate). Survey questions and responses are as follows:

1. Do you use any of the SW-846 methods listed in NR219 for WPDES monitoring?
   - Yes – 6 respondents
   - No – 13 respondents.

2. Which SW-846 methods do you use?
   - Respondents indicated an array of SW-846 methodologies, the majority of which were related to metals, volatile organics, and semivolatile organics.

3. Anticipated costs:
   - $50
   - $1,300
   - $2,000
   - $3,000-$4,500
   - $25,000 (Note: There is no substantiation or rationale for this figure)
   - Unknown

4. Lab equipment costs
   - None/minor costs

5. Any anticipated difficulties
   - Two said they would have to run both old and new methods which would be somewhat of a burden.

6. Other costs
   - Most say none; one respondent said $42,000/year resulting from running additional controls and standards. The elimination of SW-846 methods , however, will not result in any increased frequency for quality control samples or standards.

7. Respondent classification
   - Small business: 4
   - Business: 1
   - Municipality/WWTF: 1

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# ADMINISTRATIVE RULES
## Fiscal Estimate & Economic Impact Analysis

In summary:

Minimal economic impact associated with this rule revision is anticipated. The majority of the costs are upfront costs resulting from labs having to change SOPs and from validating the new EPA methods. Even the minimal fiscal impact is unavoidable as this rule revision is being directed by the U.S. EPA.

13. Benefits of Implementing the Rule and Alternative(s) to Implementing the Rule
Wastewater analysis methods will be in compliance with EPA standards.

14. Long Range Implications of Implementing the Rule
The costs are primarily up-front implementation costs; therefore, there are very few long-range implications of implementing this rule beyond the WDNR being in compliance with EPA standards.

15. Compare With Approaches Being Used by Federal Government
These regulations are established by the U.S. EPA.

16. Compare With Approaches Being Used by Neighboring States (Illinois, Iowa, Michigan and Minnesota)
All the other U.S. EPA Region 5 states (Illinois, Indiana, Michigan, Minnesota and Ohio) are subject to the U.S. EPA regulations that are delegated to the states for implementation. Wisconsin's rules for laboratory standards should essentially be the same as the other states.

| 17. Contact Name | 18. Contact Phone Number |
|---|---|
| Richard Mealy | (608) 264-6006 |

This document can be made available in alternate formats to individuals with disabilities upon request.

STATE OF WISCONSIN
DEPARTMENT OF ADMINISTRATION
DOA-2049 (R03/2012)

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# ADMINISTRATIVE RULES
# Fiscal Estimate & Economic Impact Analysis

## ATTACHMENT A

1. Summary of Rule's Economic and Fiscal Impact on Small Businesses (Separately for each Small Business Sector, Include Implementation and Compliance Costs Expected to be Incurred)

2. Summary of the data sources used to measure the Rule's impact on Small Businesses

3. Did the agency consider the following methods to reduce the impact of the Rule on Small Businesses?

☐ Less Stringent Compliance or Reporting Requirements
☐ Less Stringent Schedules or Deadlines for Compliance or Reporting
☐ Consolidation or Simplification of Reporting Requirements
☐ Establishment of performance standards in lieu of Design or Operational Standards
☐ Exemption of Small Businesses from some or all requirements
☐ Other, describe:

4. Describe the methods incorporated into the Rule that will reduce its impact on Small Businesses

5. Describe the Rule's Enforcement Provisions

6. Did the Agency prepare a Cost Benefit Analysis (if Yes, attach to form)

☐ Yes ☐ No

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

The statement of scope for this rule, SS 032-12 was approved by the Governor on May 29, 2012, published in Register No. 678, on June 14, 2012 and approved by the Natural Resources Board on June 27, 2012.

The Wisconsin Natural Resources Board proposes an order to repeal NR 219.04, Table BM; to renumber NR 219.05 and NR 219.06; to amend NR 157.21, NR 219.04 (1), (2), (4)(Note) and NR 233.40 (2); to repeal and recreate NR 219.04 Tables A, B, C, D, E, EM, ES and F; and to create NR 219.04 Tables G and H, relating to analytical methods used for Wisconsin Pollutant Discharge Elimination System (WPDES) compliance.

SS-14-12

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Analysis Prepared by the Department of Natural Resources

**1. 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 statutes or rules:**
Chs. 157, 233, Wis. Adm. Code and Chapter 283, Stats.

**5. Plain language analysis:**
Specifically, the proposed rule package will address EPA's issues with the Department rule (NR 219) incorporating SW 846 methods for wastewater sample analysis. The EPA publication SW- 846, entitled Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, is the waste division of EPA's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with the federal Resource Conservation and Recovery Act (RCRA) regulations. The DNR has allowed SW 846 methods for analysis of wastewater samples since 1995 because the methods are revised frequently and contain stringent quality control measures. However, EPA has requested that DNR remove from NR 219 all references to allowing SW 846 methods as "approved" methods for analysis of wastewater samples. EPA has also requested that DNR update NR 219 to incorporate analytical methods that have been federally approved in the Federal Register (40 CFR 136). Therefore, one objective of this rule change is to remove analytical methods that EPA has not approved for wastewater from the list of approved analyses in NR 219. The other objective is to add methods that are currently allowed by EPA per 40 CFR Part 136 but are not included in NR 219 at this time.

**6. Summary and comparison with existing and proposed federal regulations.**

The federal counterpart to this rule is 40 CFR 136. On May 18, 2012, EPA published as final rule, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures". The proposed revision to ch. NR 219 incorporates these changes and is consistent with the federal regulations.

**7. Comparison of similar 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. Wisconsin has been the only state within EPA Region 5 which has allowed the use of SW-846 methods for NPDES compliance monitoring. Therefore, removing SW-846 methods from the rule will bring Wisconsin into line with other states.

With respect to new methods promulgated by the EPA, the Illinois EPA incorporated changes to 40 CFR Part 136 with legislation which took effect February 2013. The State of Michigan does not certify laboratories that perform analyses of wastewater. The Minnesota Environmental Laboratory Accreditation Program incorporated the changes to 40 CFR Part 136 for its October 2013 application period. The Iowa Department of Natural Resources rules reference the 2007 edition of 40 CFR Part 136 at this time.

**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 an economic impact analysis:**

Alternate approved methods from different authoritative sources are available for all of the analytical methods that are being deleted. 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.

**10. Effect on small business:**

The proposed changes to ch. NR 219 potentially only affect larger certified commercial laboratories and larger municipal wastewater facilities that analyze samples for compliance with their Wisconsin Pollutant Discharge Elimination System (WPDES) permit. There are 371 laboratories certified or registered to perform analyses by the department. Of these, 238 municipal wastewater laboratories, 48 industrial environmental, and 71 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 371 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. A copy of any comments and opinion prepared by the Board of Veterans Affairs under s. 45.03 (2m), Stats., for rules proposed by the Department of Veterans Affairs:**
Not applicable

**12. Agency Contact (include email and telephone number):**
Richard Mealy, Program Chemist
Richard.Mealy@Wisconsin.gov
(608) 264-6006

**13. Place where comments are to be submitted and deadline for submission.**
Written comments may be submitted at the public hearings, by regular mail, by fax or e-mail:

101 S Webster St (use this for overnight deliveries)
PO Box 7921
Madison WI 53707-7921
Program fax: 608-266-5226
Program email: DNRLabCert@Wisconsin.gov

Written comments may also be submitted to the Department using the Wisonsin Administrative Rules Internet Web site at http://adminrules.wisconsin.gov.

Hearing dates and the comment submission deadline are to be determined.

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SECTION 1. NR 157.21 and NR 233.40(2) are amended to read:

**NR 157.21 Approval of alternate test procedures**. Applications for approval of alternate test procedures for wastewater analysis must be made as directed in s. ~~NR 219.05~~ 219.033.

**NR 233.40 (2)** Except as provided in s. ~~NR 219.05~~ 219.033, the discharge parameter values required under the clean water act shall be determined by one of the analytical methods cited in Table 7.

SECTION 2. NR 219.02 (1) and (2) are amended to read:

**NR 219.02 Applicability.** (1) The procedures prescribed herein shall, except as provided in s. NR ~~219.06~~ 219.037, be used in the determination of concentrations and quantities of pollutant parameters

as required for:

(a) An application submitted to the department for a permit under ch. 283, Stats.

(b) Reports required to be submitted by dischargers in accordance with the conditions of issued permits.

**NR 219.02** (**2)** Section NR ~~219.05~~ 219.037 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149.

SECTION 3. NR 219.05 and 219.06 are renumbered NR 219.033 and NR 219.037.

SECTION 4. NR 219.04 (1), (2) and (4)(Note) are amended to read:

**NR 219.04 Identification of test procedures.** (1) ANALYTICAL TEST PROCEDURES. Parameters or pollutants, for which wastewater analytical methods are approved, are listed together with test procedure descriptions and references in tables A to ~~E~~ H. Parameters or pollutants, for which sludge analytical methods are approved, are listed together with test procedure descriptions and references in table EM. ~~Metals samples digestion procedures and references are listed in table BM.~~ The discharge values for the listed parameters shall be determined by one of the standard analytical test procedures identified in a table under this subsection or by an alternate test procedure established under ss. ~~NR 219.05~~ 219.033 and 149.12.

**NR 219.04 (2)** Sample preservation procedures. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to ~~E~~H are prescribed in table F. Sludge samples shall be preserved at the time of collection by cooling to less than or equal to 6° C where required. All samples requiring thermal preservation at 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.

**NR 219.04 (4) Note:** Copies of the publications referenced in Tables A−~~F~~ H are available for inspection at the offices of the department of natural resources and the legislative reference bureau. Many of these materials are also available through inter−library loan.

SECTION 5: NR 219.04 Table A is repealed and recreated to read:

**TABLE A—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE**

| Parameter and units | Analytical Technology [1] | EPA | Standard methods | 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 | p. 132 [3]<br>1680 [11,15]<br>1681 [11,20] | 9221 C-2006<br>9221 E-2006 | | |
| | Membrane filter (MF) [2], single step | p. 124 [3] | 9222 D-1997 | B-0050-85 [4] | |
| 2. Coliform (fecal) in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 132 [3] | 9221 C-2006<br>9221 E-2006 | | |
| | MF [2], single step [5] | p. 124 [3] | 9222 D-1997 | | |
| 3. Coliform (total), number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 [3] | 9221 B-2006 | | |
| | MF [2], single step or two step | p. 108 [3] | 9222 B-1997 | B-0025-85 [4] | |
| 4. Coliform (total), in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 [3] | 9221 B-2006 | | |
| | MF [2] with enrichment [5] | p. 111 [3] | 9222 (B + B.5c)−1997 | | |
| 5. *E. coli*, number per 100 mL [21] | MPN [6,8,16] multiple tube, or | | 9221B.1-2006/ 9221F-2006 [12,14] | | |
| | multiple tube/multiple well, or | | 9223 B-200 4[13] | 991.15 [10] | Colilert® [13, 18]<br>Colilert-18® [13,17,18] |
| | MF [2,6,7,8] single step | 1603 [22] | | | mColiBlue ®[19] |
| 6. Fecal streptococci, number per 100 mL | MPN, 5 tube 3 dilution, or | p. 139 [3] | 9230 B-2007 | | |
| | MF [2], or | p. 136 [3] | 9230 C-2007 | B-0055-85 [4] | |
| | Plate count | p. 143 [3] | | | |
| 7. Enterococci, number per 100 mL [22] | MPN [6, 8], multiple tube/multiple well, or | | | D6503-99 [9] | Enterolert®[13, 24] |
| | MF [2, 6, 7, 8] single step or | 1600 [25] | 9230 C-2007 | | |
| | Plate count | p. 143 [3] | | | |
| 8. *Salmonella*, number per gram dry weight [11] | MPN multiple tube | 1682 [23] | | | |

**Aquatic Toxicity:**

| | | |
|---|---|---|
| 9. Toxicity, acute, fresh water organisms, $LC_{50}$, percent effluent | *Ceriodaphnia dubia* acute | 2002.0 [26] |
| | *Daphnia puplex*and*Daphnia magna* acute | 2021.0 [26] |
| | Fathead Minnow,*Pimephales promelas,*and Bannerfin shiner,*Cyprinella leedsi,*acute | 2000.0 [26] |
| | Rainbow Trout,*Oncorhynchus mykiss,*and brook trout,*Salvelinus fontinalis,*acute | 2019.0 [26] |
| 10. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, $LC_{50}$, percent effluent | Mysid,*Mysidopsis bahia,*acute | 2007.0 [26] |
| | Sheepshead Minnow,*Cyprinodon variegatus,*acute | 2004.0 [26] |
| | Silverside,*Menidia beryllina, Menidia menidia,*and*Menidia peninsulae,*acute | 2006.0 [26] |
| 11. Toxicity, chronic, fresh water organisms, NOEC or $IC_{25}$, percent effluent | Fathead minnow,*Pimephales promelas,*larval survival and growth | 1000.0 [27] |
| | Fathead minnow,*Pimephales promelas,*embryo-larval survival and teratogenicity | 1001.0 [27] |
| | Daphnia,*Ceriodaphnia dubia,*survival and reproduction | 1002.0 [27] |
| | Green alga,*Selenastrum capricornutum,*growth | 1003.0 [27] |
| 12. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or $IC_{25}$, percent effluent | Sheepshead minnow,*Cyprinodon variegatus,*larval survival and growth | 1004.0 [28] |
| | Sheepshead minnow,*Cyprinodon variegatus,*embryo-larval survival and teratogenicity | 1005.0 [28] |
| | Inland silverside,*Menidia beryllina,*larval survival and growth | 1006.0 [28] |

| | |
|---|---|
| Mysid,*Mysidopsis bahia,*survival, growth, and fecundity | 1007.0 [28] |
| Sea urchin,*Arbacia punctulata,*fertilization | 1008.0 [28] |

[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] Microbiological Methods for Monitoring the Environment, Water, and Wastes, EPA/600/8-78/017. 1978. US EPA.

[4] 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. 1989. USGS.

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

[6] 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.

[7] 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.

[8] 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.

[9] Annual Book of ASTM Standards-Water and Environmental Technology, Section 11.02. 2000, 1999, 1996. ASTM International.

[10] Official Methods of Analysis of AOAC International. 16th Edition, 4th Revision, 1998. AOAC International.

[11] Recommended for enumeration of target organism in sewage sludge.[12] The multiple-tube fermentation test is used in 9221B.1-2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB 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.

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

[14] After prior enrichment in a presumptive medium for total coliform using 9221B.1-2006, 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-2006. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

[15] Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium, EPA-821-R-10-003. April 2010. U.S. EPA.

[16] Samples shall be enumerated by the multiple-tube or multiple-well 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®, Quanti-Tray®/2000, and the MPN calculated from the table provided by the manufacturer.

[17] 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.

[18] 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.

[19] A description of the mColiBlue24® test, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

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

[21] Recommended for enumeration of target organism in wastewater effluent.

[22] Method 1603:*Escherichia coli*(*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant*Escherichia coli*Agar (modified mTEC), EPA-821-R-09-007. December 2009. U.S. EPA. U.S. Environmental Protection Agency, Office of Water, Washington, DC

[23] Method 1682:*Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium, EPA-821-R-06-014. July 2006. U.S. EPA.

[24] A description of the Enterolert® test may be obtained from IDEXX Laboratories Inc

[25] Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA-821-R-09-016. December 2009. U.S. EPA.

[26] Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. EPA-821-R-02-012. Fifth Edition, October 2002. U.S. EPA.

[27] Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. EPA-821-R-02-013. Fourth Edition, October 2002. U.S. EPA.

[28] Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. EPA-821-R-02-014. Third Edition, October 2002. U.S. EPA.

[29] Standard Methods for the Analysis of Water and Wastewater. With the promulgation of Federal Register /Vol. 77, No. 97 / Friday, May 18, 2012, the EPA lists only the most recently EPA-approved version of a Standard Method (regardless of the printed or online edition) in 40 CFR Part 136, with few exceptions, to identify the method with the year of Standard Methods approval or adoption designated by the last four digits in the method number (e.g., Standard Method 3113B–2004). This approach clearly identifies the version of the standard method approved under Part 136 and no longer ties it to a particular compendium printing or edition of Standard Methods. Methods can be purchased at www.standardmethods.org/.

SECTION 6: NR 219.04 Table B is repealed and recreated to read:

**TABLE B—LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER**

| Parameter, Units | Analytical Technology [58] | EPA [52] | Standard methods | ASTM | USGS AOAC Other |
|---|---|---|---|---|---|
| 1. Acidity, as $CaCO_3$, mg/L | Electrometric endpoint or phenolphthalein endpoint | | 2310 B-1997 | D1067-06 | I-1020-85 [2] |
| 2. Alkalinity, as $CaCO_3$, mg/L | Electrometric or Colorimetric titration to pH 4.5, Manual | | 2320 B-1997 | D1067-06 | I-1030-85 [2]<br>973.43 [3] |
| | Automatic | 310.2 (Rev. 1974)[1] | | | I-2030-85 [2] |
| 3. Aluminum—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 D-1999 or 3111 E-1999 | | I-3051-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | |
| | Stabilized temperature graphite furnace AA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP-AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3]<br>I-4471-97 [50] |
| | Direct Current Plasma (DCP) [36] | | | D4190-08 | Note [54] |
| | Colorimetric (Eriochrome cyanine R) | | 3500-Al B-2001 | | |
| 4. Ammonia (as N), mg/L | Manual distillation [6] or gas diffusion (pH > 11), followed by any of the following: | 350.1, Rev. 2.0 (1993) | 4500-$NH_3$B-1997 | | 973.49 [3] |
| | Titration | | 4500-$NH_3$C-1997 | | |
| | Electrode | | 4500-$NH_3$D-1997 or E-1997 | D1426-08 (B) | |
| | Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods | | 4500-$NH_3$F-1997 | | Note [60] |

| | | | | | |
|---|---|---|---|---|---|
| | Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods | 350.1[30], Rev. 2.0 (1993) | 4500-$NH_3$G-1997 4500-$NH_3$H-1997. | | I-4523-85 [2] |
| | Automated electrode | | | | Note [7] |
| | Ion Chromatography | | | D6919-09 | |
| 5. Antimony—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| 6. Arsenic-Total,[4] mg/L | Digestion,[4] followed by any of the following: | 206.5 (Issued 1978) [1] | | | |
| | AA gaseous hydride | | 3114 B-2009 or 3114 C-2009 | D2972-08 (B) | I-3062-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D2972-08 (C) | I-4063-98 [49] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4020-05 [70] |

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|---|---|---|---|---|---|
| | Colorimetric (SDDC) | | 3500-As B-1997 | D2972-08 (A) | I-3060-85 [2] |
| 7. Barium-Total,[4] mg/L | Digestion[4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 D-1999 | | I-3084-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D4382-02(07) | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) [36] | | | | Note [34] |
| 8. Beryllium—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D-1999 or 3111 E-1999 | D3645-08 (A) | I-3095-85.[2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3645-08 (B) | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) | | | D4190-08 | Note [34] |
| | Colorimetric (Aluminon) | | Note [61] | | |
| 9. Biochemical oxygen demand (BOD5), mg/L | Dissolved Oxygen Depletion | | 5210 B-2001 | | 973.44 [3], p. 17 [9], I-1578-78 [8] Notes [10,63] |

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|---|---|---|---|---|---|
| 10. Boron—Total,[37] mg/L | Colorimetric (Curcumin) | | 4500-B B - 2000 | | I-3112-85 [2] |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) | | | D4190-08 | Note [54] |
| 11. Bromide, mg/L | Titrimetric | | | | I-1125-85 [2] |
| | Ion selective electrode (ISE) | | | D1246-05 | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) 300.1-1, Rev 1.0 (1997) | 4110 B-2000, C-2000, D-2000 | D4327-03 | 993.30 [3] |
| | CIE/UV | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [54] |
| 12. Cadmium—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 or 3111 C-1999 | D3557-02(07) (A or B) | 974.27 [3], p. 37 [9], I-3135-85 [2] or I-3136-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3557-02(07) (D) | I-4138-89 [51] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-1472-85 [2] or I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [54] |

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|---|---|---|---|---|---|
| | Voltametry [11] | | | D3557-02(07) (C) | |
| | Colorimetric (Dithizone) | | 3500-Cd-D-1990 | | |
| 13. Calcium—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | D511-08(B) | I-3152-85 [2] |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| | Direct current plasma (DCP) | | | | Note [54] |
| | Titrimetric (EDTA) | | 3500-Ca B-1997 | D511-08 (A) | |
| | Ion Chromatography | | | D6919-09 | |
| 14. Carbonaceous biochemical oxygen demand ($CBOD_5$), mg/L[12] | Dissolved Oxygen Depletion with nitrification inhibitor | | 5210 B-2001 | | Note [55,65] |
| 15. Chemical oxygen demand (COD), mg/L | Titrimetric | 410.3 (Rev. 1978)[1] | 5220 B-1997 or C-1997 | D1252-06 (A) | I-3560-85 [2], 973.46 [3], p. 17 [9] |
| | Spectrophotometric, manual or automatic | 410.4, Rev. 2.0 (1993) | 5220 D-1997 | D1252-06 (B) | Note [13,14] I-3561-85.[2] |
| 16. Chloride, mg/L | Titrimetric: (silver nitrate) | | 4500-$Cl^-$ B-1997 | D512-04 (B) | I-1183-85 [2] |
| | Colorimetric: manual | | | | I-1187-85 [2] |
| | Colorimetric, Automated (Ferricyanide) | | 4500-$Cl^-$ E-1997 | | I-2187-85 [2] |
| | Potentiometric Titration | | 4500-$Cl^-$ D-1997 | | |

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| | Ion Selective Electrode | | | D512-04 (C) | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or 4110 C-2000 | D4327-03 | 993.30 [3], I-2057-90 [51] |
| | Capillary ion electrophoresis ( CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [54] |
| 17. Chlorine-Total residual, mg/L | Amperometric direct | | 4500-Cl D-2000 | D1253-08 | |
| | Amperometric direct (low level) | | 4500-Cl E-2000 | | |
| | Iodometric direct | | 4500-Cl B-2000 | | |
| | Back titration ether end-point[15] | | 4500-Cl C-2000 | | |
| | Colorimetric, DPD-FAS | | 4500-Cl F-2000 | | |
| | Spectrophotometric, DPD | | 4500-Cl G-2000 | | |
| | Ion selective electrode (ISE) | | | | Note [16] |
| 17A. Chlorine-Free Available, mg/L | Amperometric direct | | 4500-Cl D-2000 | D1253-08 | |
| | Amperometric direct (low level) | | 4500-Cl E-2000 | | |
| | DPD-FAS | | 4500-Cl F-2000 | | |
| | Spectrophotometric, DPD | | 4500-Cl G-2000 | | |
| 18. Chromium VI dissolved, mg/L | 0.45-micron Filtration followed by any of the following: | | | | |
| | AA chelation-extraction | | 3111 C-1999 | | I-1232-85 [2] |
| | Ion Chromatography | 218.6, Rev. | 3500-Cr C-2009 | D5257-03 | 993.23 |

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|---|---|---|---|---|---|
| | | 3.3 (1994) | | | |
| | Colorimetric (Diphenyl-carbazide) | | 3500-Cr B-2009 | D1687-02(07) (A) | I-1230-85 [2] |
| 19. Chromium—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 | D1687-02(07) (B) | 974.27,[3] I-3236-85 [2] |
| | AA chelation-extraction | | 3111 C-1999 | | |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D1687-02(07) (C) | I-3233-93 [46] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003),[68] 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[5] I-4020-05 [70] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [54] |
| | Colorimetric (Diphenyl-carbazide) | | 3500-Cr B-2009 | | |
| 20. Cobalt—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 or 3111 C-1999 | D3558-08 (A or B) | p. 37,[9] I-3239-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3558-08 (C) | I-4243-89 [51] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission | 200.5, Rev 4.2 (2003) [68]; | 3120 B-1999 | D1976-07 | I-4471-97 [50] |

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|---|---|---|---|---|---|
| | spectrometry (ICP/AES) [36] | 200.7, Rev. 4.4 (1994) | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4020-05 [70] |
| | Direct current plasma (DCP) | | | D4190-08 | Note [54] |
| 21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity | Colorimetric (ADMI) | | | | Note [18] |
| | Colorimetric (Platinum cobalt) | | 2120 B-2001 | | I-1250-85 [2] |
| 22. Copper—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 or 3111 C-1999 | D1688-07 (A or B) | 974.27,[3] p. 37,[9] I-3270-85 [2] or I-3271-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D1688-07 (C) | I-4274-89 [51] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4020-05 [70] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [54] |
| | Colorimetric (Neocuproine) | | 3500-Cu B-1999 | | |
| | Colorimetric (Bathocuproine) | | 3500-Cu C-1999 | | Note [19] |
| 23. Cyanide—Total, mg/L | Automated UV digestion/distillation and Colorimetry | | | | Kelada-01.[55] |
| | Segmented Flow Injection, In- | | | D7511-09 | |

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|---|---|---|---|---|---|
| | Line Ultraviolet Digestion, followed by gas diffusion amperometry | | | | |
| | Manual distillation with $MgCl_2$, followed by any of the following: | 335.4, Rev. 1.0 (1993) [57] | 4500-CN⁻B-1999 or C-1999 | D2036-09(A), D7284-08 | 10-204-00-1-X [56] |
| | Flow Injection, gas diffusion amperometry | | | D2036-09(A) D7284-08 | |
| | Titrimetric | | 4500-CN⁻D-1999 | D2036-09(A) | p. 22 [9] |
| | Colorimetry; Spectrophotometric, manual | | 4500-CN⁻E-1999 | D2036-09(A) | I-3300-85 [2] |
| | Colorimetry; Semi-Automated [20] | 335.4, Rev. 1.0 (1993) [57] | | | 10-204-00-1-X,[56] I-4302-85 [2] |
| | Ion Chromatography | | | D2036-09(A) | |
| | Ion Selective Electrode | | 4500-CN⁻F-1999 | D2036-09(A) | |
| 24. Cyanide-Available, mg/L | Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$, followed by Titrimetric or Spectrophotometric | | 4500-CN⁻G-1999 | D2036-09(B) | |
| | Flow injection and ligand exchange, followed by gas diffusion amperometry [59] | | | D6888-09 | OIA-1677-09 [44] |
| | Automated Distillation and Colorimetry (no UV digestion) | | | | Kelada-01 [55] |
| 24.A Cyanide-Free, mg/L | Flow Injection, followed by gas diffusion amperometry | | | D7237-10 | OIA-1677-09 [44] |
| | Manual micro-diffusion and colorimetry | | | D4282-02 | |
| 25. Fluoride—Total, mg/L | Manual distillation,[6] followed by any of the following: | | 4500-F⁻B-1997 | | |
| | Electrode, manual (ISE) | | 4500-F⁻C-1997 | D1179-04 (B) | |

| | | | | | |
|---|---|---|---|---|---|
| | Electrode, automated (ISE) | | | | I-4327-85 [2] |
| | Colorimetric, (SPADNS) | | 4500-$F^-$D-1997 | D1179-04 (A) | |
| | Automated complexone | | 4500-$F^-$E-1997 | | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and<br>300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 [3] |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [3,4] |
| 26. Gold—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 231.2 (Issued 1978)[1] | 3113 B-2004 | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| | Direct current plasma (DCP) | | | | Note [3,4] |
| 27. Hardness—Total, as $CaCO_3$, mg/L | Automated colorimetric | 130.1 (Issued 1971)[1] | | | |
| | Titrimetric (EDTA) | | 2340 C-1997 | D1126-02(07) | 973.52B,[3] I-1338-85 [2] |
| | Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33). | | 2340 B-1997 | | |
| 28. Hydrogen ion (pH), pH units | Electrometric measurement | | 4500-$H^+$B-2000 | D1293-99 (A or B) | 973.41,[3] I-1586-85 [2] |
| | Automated electrode | 150.2 (Dec. 1982)[1] | | | See footnote,[21] I-2587-85 [2] |
| 29. Iridium—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |

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|---|---|---|---|---|---|
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 235.2 (Issued 1978)[1] | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | | 3125 B-2009 | | |
| 30. Iron—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 or 3111 C-1999 | D1068-05 (A or B) | 974.27,[3] I-3381-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D1068-05 (C) | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14.[3] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [34] |
| | Colorimetric (Phenanthroline) | | 3500-Fe B-1997 | D1068-05 (D) | Note [22] |
| 31. Kjeldahl Nitrogen [5]—Total, (as N), mg/L | Manual digestion [20] and distillation or gas diffusion, followed by any of the following: | | 4500-$N_{org}$B-1997 or C-1997 and 4500-$NH_3$B-1997 | D3590-02(06) (A) | I-4515-91 [45] |
| | Titration | | 4500-$NH_3$C-1997 | | 973.48 [3] |
| | Electrode | | 4500-$NH_3$D-1997 or E-1997 | D1426-08 (B) | |
| | Semi-automated phenate | 350.1 Rev 2.0 | 4500- | | |

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|---|---|---|---|---|---|
| | | 1993 | $NH_3G$-1997. 4500-$NH_3H$-1997 | | |
| | Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods | | 4500-$NH_3F$-1997 | | Note [60] |
| **Automated Methods for TKN that do not require manual distillation** | | | | | |
| | Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation) | 351.1 (Rev. 1978)[1] | | | I-4551-78.[8] |
| | Semi-automated block digestor colorimetric (distillation not required) | 351.2, Rev. 2.0 (1993) | 4500-$N_{org}$D-1997 | D3590-02(06) (B) | I-4515-91 [45] |
| | Block digester, followed by Auto distillation and Titration | | | | Note [39] |
| | Block Digester, followed by Flow injection gas diffusion (distillation not required) | | | | Note [41] |
| 32. Lead—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 or 3111 C-1999. | D3559-08 (A or B) | 974.27,[3] I-3399-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3559-08 (D) | I-4403-89 [51] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [34] |

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|---|---|---|---|---|---|
| | Voltametry[11] | | | D3559-08 (C) | |
| | Colorimetric (Dithizone) | | 3500-Pb B-1997 | | |
| 33. Magnesium—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | D511-08 (B) | 974.27,[3] I-3447-85 [2] |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| | Direct current plasma (DCP) | | | | Note [54] |
| | Ion Chromatography | | | D6919-09 | |
| 34. Manganese—Total [4], mg/L | Digestion [4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 | D858-07 (A or B) | 974.27,[3] I-3454-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D858-07 (C) | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [54] |
| | Colorimetric (Persulfate) | | 3500-Mn B-1999 | | 920.203 [3] |

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|---|---|---|---|---|---|
| | Colorimetric (Periodate) | | | | Note [23] |
| 35. Mercury—Total,[4] mg/L | Cold vapor, Manual | 245.1, Rev. 3.0 (1994) | 3112 B-2009 | D3223-02(07) | 977.22,[3] I-3462-85 [2] |
| | Cold vapor, Automated | 245.2 (Issued 1974)[1] | | | |
| | Cold vapor atomic fluorescence spectrometry (CVAFS) | 245.7 Rev. 2.0 (2005)[17] | | | I-4464-01 [71] |
| | Purge and Trap CVAFS | 1631E[43] | | | |
| 36. Molybdenum—Total,[4] mg/L | Digestion,[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D-1999 | | I-3490-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | I-3492-96 [47] |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4471-97 [50] |
| | Direct current plasma (DCP) | | | | Note [34] |
| 37. Nickel—Total,[4] mg/L | Digestion [4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 or 3111 C-1999 | D1886-08 (A or B) | I-3499-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D1886-08 (C) | I-4503-89 [51] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) [36] | 200.5, Rev 4.2 (2003) [68]; 200.7, Rev. | 3120 B-1999 | D1976-07 | I-4471-97 [50] |

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|---|---|---|---|---|---|
| | | 4.4 (1994) | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14,[3] I-4020-05 [70] |
| | Direct current plasma (DCP) [36] | | | D4190-08 | Note [34] |
| 38. Nitrate (as N), mg/L | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 [3] |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [54] |
| | Ion Selective Electrode | | 4500-$NO_3^-$D-2000 | | |
| | Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40) | | | | Note [62] |
| 39. Nitrate + nitrite (as N), mg/L | Cadmium reduction, Manual | | 4500-$NO_3^-$E-2000 | D3867-04 (B) | |
| | Cadmium reduction, Automated | 353.2, Rev. 2.0 (1993) | 4500-$NO_3^-$F-2000 | D3867-04 (A) | I-2545-90 [51] |
| | Automated hydrazine | | 4500-$NO_3^-$H-2000 | | |
| | Reduction/Colorimetric | | | | Note [62] |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 [3] |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [54] |
| 40. Nitrite (as N), mg/L | Spectrophotometric: Manual | | 4500-$NO_2^-$B-2000 | | Note [25] |
| | Automated (Diazotization) | | | | I-4540-85[2], Note [62] |
| | Automated (*bypass cadmium reduction) | 353.2, Rev. 2.0 (1993) | 4500-$NO_3^-$F-2000 | D3867-04 (A) | I-4545-85 [2] |

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|---|---|---|---|---|---|
| | Manual (*bypass cadmium reduction) | | 4500-$NO_3^-$E-2000 | D3867-04 (B) | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 [3] |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [3,4] |
| 41. Oil and grease—Total recoverable, mg/L | Hexane extractable material (HEM): n-Hexane extraction and gravimetry | 1664 Rev. A; 1664 Rev. B [42] | 5520 B-2001 [38] | | |
| | Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry | 1664 Rev. A; 1664 Rev. B[42] | 5520 B-2001[38]and 5520 F-2001[38] | | |
| 42. Organic carbon—Total (TOC), mg/L | Combustion | | 5310 B-2000 | D7573-09 | 973.47[3], p. 14 [24] |
| | Heated persulfate or UV persulfate oxidation | | 5310 C 2000 5310 D 2000. | D4839-03 | 973.47[3], p. 14 [24] |
| 43. Organic nitrogen (as N), mg/L | Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4) | | | | |
| 44. Ortho-phosphate (as P), mg/L | Colorimetry, Ascorbic acid, Automated | 365.1, Rev. 2.0 (1993) | 4500-P F-1999 or G-1999 | | 973.56[3], I-4601-85 [2] |
| | Colorimetry, Ascorbic Acid, Manual single reagent | | 4500-P E-1999 | D515-88(A) | 973.55 [3] |
| | Colorimetry, Ascorbic Acid, Manual two reagent | 365.3 (Issued 1978)[1] | | | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 [3] |
| | Capillary ion electrophoresis ( CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [3,4] |
| 45. Osmium—Total[4], mg/L | Digestion[4], followed by any of the following: | | | | |

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|---|---|---|---|---|---|
| | AA direct aspiration (FLAA) | | 3111 D-1999 | | |
| | Graphite furnace AA (GFAA) | 252.2 (Issued 1978)[1] | | | |
| 46. Oxygen, dissolved, mg/L | Winkler (Azide modification) | | 4500-O B-2001, C-2001, D-2001, E-2001, F-2001 | D888-09 (A) | 973.45B [3], I-1575-78 [8] |
| | Electrode | | 4500-O G-2001 | D888-09 (B) | I-1576-78 [8] |
| | Luminescence Based Sensor | | | D888-09 (C) | Note [63] Note [64] |
| 47. Palladium—Total,[4] mg/L | Digestion [4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 253.2[1](Issued 1978) | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | | 3125 B-2009 | | |
| | Direct current plasma (DCP) | | | | Note [34] |
| 48. Phenols, mg/L | Manual distillation[26], followed by any of the following: | 420.1[1](Rev. 1978) | 5530 B-2005 | D1783-01 | |
| | Colorimetric (4AAP) manual | 420.1[1](Rev. 1978) | 5530 D-2005[27] | D1783-01 (A or B) | |
| | Colorimetric (4AAP), Automated | 420.4 Rev. 1.0 (1993) | | | |
| 49. Phosphorus (elemental), mg/L | Gas-liquid chromatography | | | | Note [28] |
| 50. Phosphorus—Total, mg/L | Digestion[20], followed by any of the following: | | 4500-P B(5)-1999 | | 973.55 [3] |
| | Colorimetric, Manual ascorbic acid | 365.3[1](Issued 1978) | 4500-P E-1999 | D515-88 (A) | |
| | Colorimetric, Automated ascorbic acid reduction | 365.1 Rev. 2.0 (1993) | 4500-P F-1999, G-1999, H-1999 | | 973.56 [3], I-4600-85 [2] |

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|---|---|---|---|---|---|
| | Colorimetric, Semi-automated block digestor (TKP digestion) | 365.4[1](Issued 1974) | | D515-88 (B) | I-4610-91 [48] |
| 51. Platinum—Total,[4]mg/L | Digestion [4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 255.2 (Issued 1978)[1] | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | | 3125 B-2009 | | |
| | Direct current plasma (DCP) | | | | Note [34] |
| 52. Potassium—Total,[4]mg/L | Digestion [4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | 973.53 [3], I-3630-85 [2] |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994) | 3120 B-1999 | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| | Flame photometric | | 3500-K B-1997 | | |
| | Electrode | | 3500-K C-1997 | | |
| | Ion Chromatography | | | D6919-09 | |
| 53. Residue—Total, mg/L | Gravimetric, 103-105°C | | 2540 B-1997 | | I-3750-85 [2] |
| 54. Residue—filterable (TDS), mg/L | Gravimetric, 180°C | | 2540 C-1997 | D5907-03 | I-1750-85 [2] |
| 55. Residue—non-filterable (TSS), mg/L | Gravimetric, 103-105°C post washing of residue | | 2540 D-1997 | D5907-03 | I-3765-85 [2] |
| 56. Residue—settleable, mg/L | Volumetric, (Imhoff cone), or gravimetric | | 2540 F-1997 | | |
| 57. Residue—Volatile, mg/L | Gravimetric, 550°C | 160.4 (Issued 1971)[1] | 2540-E-1997 | | I-3753-85 [2] |

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|---|---|---|---|---|---|
| 58. Rhodium—Total,[4] mg/L | Digestion [4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA), or | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 265.2 (Issued 1978)[1] | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | | 3125 B-2009 | | |
| 59. Ruthenium—Total,[4]mg/L | Digestion[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA), or | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 267.2[1] | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8 | 3125 B-2009 | | |
| 60. Selenium—Total,[4]mg/L | Digestion[4], followed by any of the following: | | | | |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3859-08 (B) | I-4668-98 [49] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES)[36] | 200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3], I-4020-05 [70] |
| | AA gaseous hydride | | 3114 B-2009, or 3111 C-2009 | D3859-08 (A) | I-3667-85 [2] |
| 61. Silica—Dissolved,[37]mg/L | 0.45-micron filtration followed by any of the following: | | | | |
| | Colorimetric, Manual | | 4500-$SiO_2$C-1997 | D859-05 | I-1700-85 [2] |
| | Colorimetric, Automated (Molybdosilicate) | | 4500-$SiO_2$E-1997 or F-1997 | | I-2700-85 [2] |
| | Inductively coupled | 200.5, Rev 4.2 | 3120 B- | | I-4471-97 [50] |

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|---|---|---|---|---|---|
| | plasma−atomic emission spectrometry (ICP/AES) | (2003)[68]; 200.7, Rev. 4.4 (1994) | 1999 | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| 62. Silver—Total,[4, 31] mg/L | Digestion[4, 29], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 or 3111 C-1999 | | 974.27 [3], p. 37 [9], I-3720-85 [2] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | I-4724-89 [51] |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3], I-4471-97 [50] |
| | Direct current plasma (DCP) | | | | Note [54] |
| 63. Sodium—Total,[4] mg/L | Digestion [4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | 973.54 [3], I-3735-85 [2] |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| | Direct current plasma (DCP) | | | | Note [54] |
| | Flame photometric | | 3500-Na B-1997 | | |
| | Ion Chromatography | | | D6919-09 | |
| 64. Specific conductance, micromhos/cm at 25 °C | Wheatstone bridge | 120.1[1](Rev. 1982) | 2510 B-1997 | D1125-95(99) (A) | 973.40 [3], I-2781-85 [2] |

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|---|---|---|---|---|---|
| 65. Sulfate (as $SO_4$), mg/L | Colorimetric, Automated | 375.2, Rev. 2.0 (1993) | 4500-$SO_4^{2-}$ F-1997 or G-1997 | | |
| | Gravimetric | | 4500-$SO_4^{2-}$ C-1997 or D-1997 | | 925.54 [3] |
| | Turbidimetric | | 4500-$SO_4^{2-}$ E-1997 | D516-07 | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 [3], I-4020-05 [70] |
| | Capillary ion electrophoresis ( CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 [54] |
| 66. Sulfide (as S), mg/L | Sample Pretreatment | | 4500-$S^{2-}$B, C-2000 | | |
| | Titrimetric (iodine) | | 4500-$S^{2-}$F-2000 | | I-3840-85 [2] |
| | Colorimetric (methylene blue) | | 4500-$S^{2-}$D-2000 | | |
| | Ion Selective Electrode | | 4500-$S^{2-}$G-2000 | D4658-08 | |
| 67. Sulfite (as $SO_3$), mg/L | Titrimetric (iodine-iodate) | | 4500-$SO_3^{2-}$B-2000 | | |
| 68. Surfactants, mg/L | Colorimetric (methylene blue) | | 5540 C-2000 | D2330-02 | |
| 69. Temperature, °C | Thermometric | | 2550 B-2000 | | Note [32] |
| 70. Thallium-Total,[4] mg/L | Digestion[4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 279.2[1](Issued 1978) | 3113 B-2004 | | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994); 200.5 Rev. 4.2 (2003)[68] | 3120 B-1999 | D1976-07 | |

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|---|---|---|---|---|---|
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3], I-4471-97 [50] |
| 71. Tin-Total,[4] mg/L | Digestion[4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | I-3850-78 [8] |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994) | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| 72. Titanium-Total,[4] mg/L | Digestion[4] followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D-1999 | | |
| | Graphite furnace AA (GFAA) | 283.2[1] (Issued 1978) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994) | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] |
| | Direct current plasma (DCP) | | | | Note [34] |
| 73. Turbidity, NTU[53] | Nephelometric | 180.1, Rev. 2.0 (1993) | 2130 B-2001 | D1889-00 | I-3860-85 [2] Note [65] Note [66] Note [67] |
| 74. Vanadium-Total,[4] mg/L | Digestion[4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D-1999 | | |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3373-03(07) | |
| | Inductively coupled plasma−atomic emission | 200.5, Rev 4.2 (2003)[68]; | 3120 B-1999 | D1976-07 | I-4471-97 [50] |

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|---|---|---|---|---|---|
| | spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994) | | | |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14[3], I-4020-05 [70] |
| | Direct current plasma (DCP) | | | D4190-08 | Note [34] |
| | Colorimetric (Gallic Acid) | | 3500-V B-1997 | | |
| 75. Zinc-Total[4], mg/L | Digestion [4], followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) [36] | | 3111 B-1999 or 3111 C-1999 | D1691-02(07) (A or B) | 974.27 [3], p. 37 [9], I-3900-85 [2] |
| | Graphite furnace AA (GFAA) | 289.2[1](Issued 1978) | | | |
| | Inductively coupled plasma−atomic emission spectrometry (ICP/AES)[36] | 200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 [50] |
| | Inductively coupled plasma−mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 [3] I-4020-05 [70] |
| | Direct current plasma (DCP)[36] | | | D4190-08 | Note [34] |
| | Colorimetric (Zincon) | | 3500 Zn B-1997 | | Note [33] |
| 76. Acid Mine Drainage | | 1627 [69] | | | |

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

[2] Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Book 5, Chapter A1., unless otherwise stated. 1989. USGS.

[3] Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International.

[4] For the determination of total metals (which are 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-platform 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 instruction 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 (platform furnace AA, 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 IB); 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.

[5] Copper sulfate or other catalysts that have been found suitable may be used in place of mercuric sulfate.

[6] 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. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step. (A total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as < 20% RPD for all tested matrices). Alternatively the two populations of spike recovery percentages may be compared using a recognized statistical test.

[7] Industrial Method Number 379-75 WE Ammonia, Automated Electrode Method, Technicon Auto Analyzer II. February 19, 1976. Bran & Luebbe Analyzing Technologies Inc.

[8] The approved method is that cited in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. USGS.

[9] American National Standard on Photographic Processing Effluents. April 2, 1975. American National Standards Institute (ANSI), 25 West 43rd St., New York, NY 10036.

[10] In-Situ Method 1003-8-2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. 2009. In-Situ Incorporated.

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

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

[13] OIC Chemical Oxygen Demand Method. 1978. Oceanography International Corporation. 512 West Loop, P.O. Box 2980, College Station, TX 77840.

[14] Method 8000, Chemical Oxygen Demand, Hach Handbook of Water Analysis, 1979. Hach Company. P.O. Box 389, Loveland, CO 80537. Avail- able on−line at http:/www.hach.com.

[15] The back titration method will be used to resolve controversy.

[16] Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70. Thermo Scientific, 81 Wyman Street, Waltham, MA 02454. 1977. Orion Research Incorporated. The calibration for the Orion residual chlorine method must be derived using at least three standard solutions, prepared from a 0.00281 N potassium iodate solution.

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

[18] National Council of the Paper Industry for Air and Stream Improvement (NCASI) Technical Bulletin 253, December 1971.

[19] Method 8506, Biocinchoninate Method for Copper, Hach Handbook of Water Analysis. 1979. Hach Company. P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.

[20] When using a method with block digestion, this treatment is not required.

[21] Industrial Method Number 378-75WA, Hydrogen ion (pH) Automated Electrode Method, Bran & Luebbe (Technicon) Autoanalyzer II. October 1976. Bran & Luebbe Analyzing Technologies. Elmsford, NY 10523.

[22] Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water. 1980. Hach Company P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.

[23] Method 8034, Periodate Oxidation Method for Manganese, Hach Handbook of Wastewater Analysis. 1979. Hach Company Loveland, CO 80537. Available on−line at http:/www.hach.com.

[24] 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, (1972 Revised 1987) p. 14. 1987. USGS. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[25] Method 8507, Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater. 1979. Hach Company P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.

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

[27] The colorimetric reaction must be conducted at a pH of 10.0 ± 0.2.

[28] Addison, R.F., and R.G. Ackman. 1970. Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography,*Journal of Chromatograph*y, 47(3):421-426. Available in most public libraries. Back volumes of the Journal of Chromatography are available from: Elsevier/North−Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

[29] 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 $Na_2S_2O_3$ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[30] The use of EDTA decreases method sensitivity. Analysts may omit EDTA or replace with another suitable complexing reagent provided that all method specified quality control acceptance criteria are met.

[31] 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 $NH_4OH$, 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 $NH_4OH$. 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.

[32] "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. USGS. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[33] Method 8009, Zincon Method for Zinc, Hach Handbook of Water Analysis, 1979. Hach Company. Loveland, CO 80537. Available on−line at http:/www.hach.com.

[34] Method AES0029, Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes. 1986-Revised 1991. Thermo Jarrell Ash Corporation. Available from: Thermo Scientific, 81 Wyman Street, Waltham, MA 02454.

[35] In-Situ Method 1004-8-2009, Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe. 2009. In-Situ Incorporated.

[36] 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.

[37] When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.

[38] Only use n-hexane (n-Hexane—85% minimum purity, 99.0% min. saturated C6 isomers, residue less than 1 mg/L) extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Methods 1664 Rev. A and 1664 Rev. B). Use of other extraction solvents is prohibited.[39] Method PAI-DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric Detection. Revised December 22, 1994. OI Analytical/ALP- KEM, P.O. Box 9010, College Station, TX 77842.

[40] Method PAI-DK02, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Colorimetric Detection. Revised December 22, 1994. OI Analytical.

[41] Method PAI-DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion. Revised December 22, 1994. OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[42] Method 1664 Rev. B is the revised version of EPA Method 1664 Rev. A. U.S. EPA. February 1999, Revision A. Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-98-002. U.S. EPA. February 2010, Revision B. Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-10-001. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.

[43] Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-02-019. Revision E. August 2002, U.S. EPA. The application of clean techniques described in EPA's 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.

[44] Method OIA-1677-09, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA). 2010. OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[45] Open File Report 00-170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. USGS.

[46] Open File Report 93-449, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1993. USGS.

[47] Open File Report 97-198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry. 1997. USGS.

[48] Open File Report 92-146, 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. 1992. USGS.

[49] Open File Report 98-639, 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. 1999. USGS.

[50] Open File Report 98-165, 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. 1998. USGS.

[51] Open File Report 93-125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.

[52] Unless otherwise indicated, all EPA methods, excluding EPA Method 300.1-1, are published in U.S. EPA. May 1994. Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111; or U.S. EPA. August 1993. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100. EPA Method 300.1 is US EPA. Revision 1.0, 1997, including errata cover sheet April 27, 1999. Determination of Inorganic Ions in Drinking Water by Ion Chromatography.

[53] Styrene divinyl benzene beads (e.g., AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StablCal™or equivalent) are acceptable substitutes for formazin.

[54] Method D6508, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. December 2000. Waters Corp. , 34 Maple St., Milford, MA, 01757, Telephone: 508/482–2131, Fax: 508/482–3625.

[55] Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate, EPA 821-B-01-009, Revision 1.2, August 2001. US EPA. 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.

[56] 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. Revision 2.2, March 2005. Lachat Instruments. Available from Hach Company, P.O. Box 389, Loveland, CO 80537.

[57] When using sulfide removal test procedures described in EPA Method 335.4-1, reconstitute particulate that is filtered with the sample prior to distillation.

[58] 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.

[59] Samples analyzed for available cyanide using OI Analytical method OIA-1677-09 or ASTM method D6888-09 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample filtration to no more than 30 minutes to preclude settling of materials in samples.

[60] Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction ("phenol-hypochlorite reaction") colorimetric ammonium determination methods. For example when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, Patton, C.J. and S.R. Crouch. March 1977. Anal. Chem. 49:464-469. These reaction parameters increase to pH > 12.6 and 665 nm when salicylate is used as the color reagent—see, Krom, M.D. April 1980. The Analyst 105:305-316.

[61] If atomic absorption or ICP instrumentation is not available, the aluminon colorimetric method detailed in the 19th Edition of *Standard Methods* may be used. This method has poorer precision and bias than the methods of choice.

[62] Easy (1-Reagent) Nitrate Method, Revision November 12, 2011. Craig Chinchilla.

[63] Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of $BOD_5$and $cBOD_5$ Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).

[64] In-Situ Method 1002-8-2009, Dissolved Oxygen (DO) Measurement by Optical Probe. 2009. In-Situ Incorporated.

[65] Mitchell Method M5331, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.

[66] Mitchell Method M5271, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.

[67] Orion Method AQ4500, Determination of Turbidity by Nephelometry. Revision 5, March 12, 2009. Thermo Scientific.

[68] EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA/600/R-06/115. Revision 4.2, October 2003. US EPA.

[69] Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality, EPA-821-R-09-002. December 2011. US EPA.

[70] Techniques and Methods Book 5-B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry, Chapter 1, Section B, Methods of the National Water Quality Laboratory, Book 5, Laboratory Analysis, 2006. USGS.

[71] Water-Resources Investigations Report 01-4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water With Cold Vapor-Atomic Fluorescence Spectrometry, 2001. USGS.

[72] 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 245.1 (manual) or 245.2 (automated). If method 1631E is modified to eliminate the purge and trap step, the appropriate method citation is 245.7.

SECTION 7: NR 219.04 Table BM is repealed.

SECTION 8: NR 219.04 Table C is repealed and recreated to read:

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

| Parameter [1] | Analytical Technology | EPA [2,7] | Standard methods | ASTM | Other |
|---|---|---|---|---|---|
| 1. Acenaphthene | GC | 610 | | | |
| | GC/MS | 625,1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 2. Acenaphthylene | GC | 610 | | | |
| | GC/MS | 625,1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 3. Acrolein | GC | 603 | | | |
| | GC/MS | 624 [4],1624B | | | |
| 4. Acrylonitrile | GC | 603 | | | |
| | GC/MS | 624 [4], 1624B | | | |
| 5. Anthracene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440B-2000 | D4657-92 (98) | |
| 6. Benzene | GC | 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 7. Benzidine | Spectro-photometric | | | | Note [3], p.1 |
| | GC/MS | 625 [5], 1625B | 6410 B-2000 | | |
| | HPLC | 605 | | | |
| 8. Benzo(a)anthracene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 9. Benzo(a)pyrene | GC | 610 | | | |

| | | | | | |
|---|---|---|---|---|---|
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 10. Benzo(b)fluoranthene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 11. Benzo(g,h,i)perylene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 12. Benzo(k)fluoranthene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 13. Benzyl chloride | GC | | | | Note [3], p. 130 |
| | GC/MS | | | | Note [6], p. S102 |
| 14. Butyl benzyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 15. bis(2-Chloroethoxy) methane | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 16. bis(2-Chloroethyl) ether | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 17. bis(2-Ethylhexyl) phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 18. Bromodichloromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 19. Bromoform | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 20. Bromomethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 21. 4-Bromophenyl phenyl ether | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 22. Carbon tetrachloride | GC | 601 | 6200 C-1997 | | Note [3], p. 130 |

| | | | | | |
|---|---|---|---|---|---|
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 23. 4-Chloro-3-methyl phenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 24. Chlorobenzene | GC | 601, 602 | 6200 C-1997 | | Note [3], p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 25. Chloroethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 26. 2-Chloroethylvinyl ether | GC | 601 | | | |
| | GC/MS | 624, 1624B | | | |
| 27. Chloroform | GC | 601 | 6200 C-1997 | | Note [3], p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 28. Chloromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 29. 2-Chloronaphthalene | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 30. 2-Chlorophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 31. 4-Chlorophenyl phenyl ether | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 32. Chrysene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 33. Dibenzo(a,h)anthracene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 34. Dibromochloromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 35. 1,2-Dichlorobenzene | GC | 601, 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1625B | 6200 B-1997 | | Note [9], p. 27 |
| 36. 1,3-Dichlorobenzene | GC | 601, 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1625B | 6200 B-1997 | | Note [9], p. 27 |

| | | | | |
|---|---|---|---|---|
| 37. 1,4-Dichlorobenzene | GC | 601, 602 | 6200 C-1997. | |
| | GC/MS | 624, 1625B | 6200 B-1997 | Note [9], p. 27 |
| 38. 3,3'-Dichlorobenzidine | GC/MS | 625, 1625B | 6410 B-2000 | |
| | HPLC | 605 | | |
| 39. Dichlorodifluoromethane | GC | 601 | | |
| | GC/MS | | 6200 C-1997 | |
| 40. 1,1-Dichloroethane | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 41. 1,2-Dichloroethane | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 42. 1,1-Dichloroethene | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 43. *trans*-1,2-Dichloroethene | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 44. 2,4-Dichlorophenol | GC | 604 | 6420 B-2000 | |
| | GC/MS | 625, 1625B | 6410 B-2000 | Note [9], p. 27 |
| 45. 1,2-Dichloropropane | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 46. *cis*-1,3-Dichloropropene | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 47. trans-1,3-Dichloropropene | GC | 601 | 6200 C-1997 | |
| | GC/MS | 624, 1624B | 6200 B-1997 | |
| 48. Diethyl phthalate | GC | 606 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | Note [9], p. 27 |
| 49. 2,4-Dimethylphenol | GC | 604 | 6420 B-2000 | |
| | GC/MS | 625, 1625B | 6410 B-2000 | Note [9], p. 27 |
| 50. Dimethyl phthalate | GC | 606 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | Note [9], p. 27 |

| | | | | | |
|---|---|---|---|---|---|
| 51. Di-n-butyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 52. Di-n-octyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 53. 2, 3-Dinitrophenol | GC | 604 | 6420 B-2000 | | Note [9], p. 27 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | |
| 54. 2, 4-Dinitrophenol | GC | 604 | 6420 B-2000 | | Note [9], p. 27 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | |
| 55. 2, 6-Dinitrophenol | GC | 604 | 6420 B-2000 | | Note [9], p. 27 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | |
| 56. 2,3-Dinitrotoluene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 57. 2,4-Dinitrotoluene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 58. 2,6-Dinitrotoluene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 59. Epichlorohydrin | GC | | | | Note [3], p. 130 |
| | GC/MS | | | | Note [6], p. S102 |
| 60. Ethylbenzene | GC | 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 61. Fluoranthene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 62. Fluorene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |

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|---|---|---|---|---|---|
| 63. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran | HRGC/MS | 1613B | | | |
| 64. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran | HRGC/MS | 1613B | | | |
| 65. 1,2,3,4,6,7,8- Heptachloro-dibenzo-p-dioxin | HRGC/MS | 1613B | | | |
| 66. Hexachlorobenzene | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 67. Hexachlorobutadiene | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 68. Hexachlorocyclopentadiene | GC | 612 | | | |
| | GC/MS | 625 [5], 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 69. 1,2,3,4,7,8-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 70. 1,2,3,6,7,8-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 71. 1,2,3,7,8,9-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 72. 2,3,4,6,7,8-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 73. 1,2,3,4,7,8-Hexachloro-dibenzo-p-dioxin | HR GC/MS | 1613B | | | |
| 74. 1,2,3,6,7,8-Hexachloro-dibenzo-p-dioxin | HR GC/MS | 1613B | | | |
| 75. 1,2,3,7,8,9-Hexachloro-dibenzo-p-dioxin | HR GC/MS | 1613B | | | |
| 76. Hexachloroethane | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 77. Indeno(1,2,3-c,d) pyrene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 78. Isophorone | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 79. Methylene chloride | GC | 601 | 6200 C-1997 | | Note [5], p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 80. 2-Methyl-4,6-dinitrophenol | GC | 604 | 6420 B-2000 | | |

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|---|---|---|---|---|---|
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 81. Naphthalene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000. | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000. | | |
| 82. Nitrobenzene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | | | D4657-92 (98) | |
| 83. 2-Nitrophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 84. 4-Nitrophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 85. N-Nitrosodimethylamine | GC | 607 | | | |
| | GC/MS | 625 [5], 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 86. N-Nitrosodi-n-propylamine | GC | 607 | | | |
| | GC/MS | 625 [5], 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 87. N-Nitrosodiphenylamine | GC | 607 | | | |
| | GC/MS | 625 [5], 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 88. Octachlorodibenzofuran | HR GC/MS | 1613B [10] | | | |
| 89. Octachlorodibenzo-p-dioxin | HR GC/MS | 1613B [10] | | | |
| 90. 2,2'-Oxybis(2-chloro-propane) [also known as bis(2-Chloroisopropyl) ether] | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 91. PCB-1016, (Aroclor or congeners)[12,13] | GC | 608 | | | Note [5], p. 43; Note [8] |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HR GC/MS | 1668A [14] | | | |
| 92. PCB-1221, (Aroclor or congeners)[12,13] | GC | 608 | | | Note [5], p. 43; Note [8] |
| | GC/MS | 625 | 6410 B-2000 | | |

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|---|---|---|---|---|---|
| | HRGC/ MS | 1668A[14] | | | |
| 93. PCB-1232, (Aroclor or congeners)[12,13] | GC | 608 | | | Note [3], p. 43; Note [8] |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A[14] | | | |
| 94. PCB-1242, (Aroclor or congeners)[12,13] | GC | 608 | | | Note [3], p. 43; Note [8] |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A[14] | | | |
| 95. PCB-1248, (Aroclor or congeners)[12,13] | GC | 608 | | | |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A[14] | | | |
| 96. PCB-1254, (Aroclor or congeners)[12,13] | GC | 608 | | | Note [3], p. 43; Note [8] |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A[14] | | | |
| 97. PCB-1260, (Aroclor or congeners)[12,13] | GC | 608 | | | Note [3], p. 43; Note [8] |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A[14] | | | |
| 98. 1,2,3,7,8-Pentachloro-dibenzofuran | GC/MS | 1613B | | | |
| 99. 2,3,4,7,8-Pentachloro-dibenzofuran | GC/MS | 1613B | | | |
| 100. 1,2,3,7,8,-Pentachloro-dibenzo-p-dioxin | GC/MS | 1613B | | | |
| 101. Pentachlorophenol | GC | 604 | 6420 B-2000 | | Note [3], p. 140 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 102. Phenanthrene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 103. Phenol | GC | 604 | 6420 B-2000 | | |

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|---|---|---|---|---|---|
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 104. Pyrene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 105. 2,3,7,8-Tetrachloro-dibenzofuran | HR GC/MS | 1613B[10] | | | |
| 106. 2,3,7,8-Tetrachloro-dibenzo-p-dioxin | GC/MS | 613, 625 [5a], | | | |
| 107. 1,1,2,2-Tetrachloroethane | GC | 601 | 6200 C-1997 | | Note [5], p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 108 Tetrachlorocatechol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 109. Tetrachloroethene | GC | 601 | 6200 C-1997 | | Note [5], p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 110. Tetrachloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 111. 2,3,4,6− Tetrachlorophenol | GC | | 6420 B-2000 | | |
| | GC/MS | | 6410 B-2000 | | |
| 112. Toluene | GC | 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 113. 1,2,4-Trichlorobenzene | GC | 612 | | | Note [5], p. 130 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9], p. 27 |
| 114. 3,4,5−Trichlorocatechol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 115. 3,4,6−Trichlorocatechol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 116. 1,1,1-Trichloroethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 117. 1,1,2-Trichloroethane | GC | 601 | 6200 C-1997 | | Note [5], p. 130 |

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|---|---|---|---|---|---|
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 118. Trichloroethene | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 119. Trichlorofluoromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624 | 6200 B-1997 | | |
| 120. 3,4,5−Trichloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 121. 3,4,6−Trichloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 122. 4,5,6−Trichloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 123. 2,4,5−Trichlorophenol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 124. 2,4,6-Trichlorophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note [9~~13~~], p. 27 |
| 125. Trichlorosyringol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653[11] | 6410 B-2000 | | |
| 126. Vinyl chloride | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 127. Nonylphenol | GC/MS | | | D7065-06 | |
| 128. Bisphenol A (BPA) | GC/MS | | | D7065-06 | |
| 129. p-tert-Octylphenol (OP) | GC/MS | | | D7065-06 | |
| 130. Nonylphenol Monoethoxylate (NP1EO) | GC/MS | | | D7065-06 | |
| 131. Nonylphenol Diethoxylate (NP2EO) | GC/MS | | | D7065-06 | |
| 132. Adsorbable Organic Halides (AOX) | Adsorption and Coulometric Titration | 1650 [11] | | | |
| 133. Chlorinated Phenolics | In Situ Acetylation and GC/MS | 1653 [11] | | | |

[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, 1613B, 1624B, and 1625B are provided at Appendix A, Test Procedures for Analysis of Organic Pollutants, of 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 40 CFR Part136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit.

[3] Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA.

[4] Method 624 may be used for quantitative determination of acrolein and acrylonitrile, provided that the laboratory has documentation to substantiate the ability to detect and quantify these analytes at levels necessary to comply with any associated regulations. In addition, the use of sample introduction techniques other than simple purge-and-trap may be required. QC acceptance criteria from Method 603 should be used when analyzing samples for acrolein and acrylonitrile in the absence of such criteria in Method 624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, N-nitrosodi-n-propylamine, 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.

[5a] Method 625, screening only.

[6] Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the 15th Edition of*Standard Methods for the Examination of Water and Wastewater.*1981. American Public Health Association (APHA).

[7] 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 in accordance with procedures each in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 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.

[8] Organochlorine Pesticides and PCBs in Wastewater Using Empore™Disk. Revised October 28, 1994. 3M Corporation.

[9] Method O-3116-87 is in Open File Report 93-125, Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.

[10] Analysts may use Fluid Management Systems, Inc. Power-Prep system in place of manual cleanup provided the analyst meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities. Method 1613, Revision B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. Revision B, 1994. U.S. EPA. The full text of this method is provided in Appendix A to 40 CFR Part 136 and at*http://water.epa.gov/scitech/methods/cwa/index.cfm.*

[11] Method 1650, Adsorbable Organic Halides by Adsorption and Coulometric Titration. Revision C, 1997. U.S. EPA. Method 1653, Chlorinated Phenolics in Wastewater by In Situ Acetylation and GCMS. Revision A, 1997. U.S. EPA. The full text for both of these methods is provided at Appendix A, “Methods 1650 and 1653”, in Part 430, The Pulp, Paper, and Paperboard Point Source Category. Also available on−line at

http:/www.gpoaccess.gov/.

[12] 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.

[13] If congener specific analysis is performed , 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, clean up steps of the extract shall be performed as necessary to remove interference. If congener specific analysis is done, clean up steps of the extract shall be performed as necessary to remove interference. If desired 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:

Florisil, Gel Permeation, Silica Gel, Alumina, Sulfur Clean Up, Sulfuric Acid Clean Up.

[14] "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: NR 219.04 Table D is repealed and recreated to read:

**TABLE D—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]**

| Parameter | Analytical Technology | EPA [2,7,10] | Standard methods | ASTM | Other |
|---|---|---|---|---|---|
| 1. Aldrin | GC | 608, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96 (02) | Note [3], p. 7;<br>Note [4], O-3104-83;<br>Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 2. Ametryn | GC | 507, 619 | | | Note [3], p. 83;<br>Note [9], O-3106-93;<br>Note [6], p. S68 |
| | GC/MS | 525.2 | | | Note [14], O-1121-91 |
| 3. Aminocarb | TLC | | | | Note [3], p. 94;<br>Note [6], p. S60 |
| | HPLC | 632 | | | |
| 4. Atraton | GC | 619 | | | Note [3], p. 83;<br>Note [6], p. S68 |
| 5. Atrazine | GC | 507, 619 | | | Note [3], p. 83;<br>Note [6], p. S68;<br>Note [9], O-3106-93 |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| | GC/MS | 525.1, 525.2 | | | Note [11], O-1126-95 |
| 6. Azinphos methyl | GC | 614, 622, 1657 | | | Note [3], p. 25;<br>Note [6], p. S51 |
| | GC-MS | | | | Note [11], O-1126-95 |
| 7. Barban | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| 8. α-BHC | GC | 608, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7;<br>Note [8], 3M0222 |
| | GC/MS | 625 [5] | 6410 B-2000 | | Note [11],<br>O-1126-95 |
| 9. β-BHC | GC | 608, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 10. δ-BHC | GC | 608, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [8],<br>3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 11. γ-BHC (Lindane) | GC | 608, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7;<br>Note [4], O-3104-83;<br>Note [8], 3M0222 |
| | GC/MS | 625 [5] | 6410 B-2000 | | Note [11], O-1126-95 |
| 12. Captan | GC | 617 | 6630 B-2000 | D3086-90, D5812-96(02) | Note [3], p. 7 |

| | | | | | |
|---|---|---|---|---|---|
| 13. Carbaryl | TLC | | | | Note [3], p. 94, Note [6], p. S60 |
| | HPLC | 531.1, 632 | | | |
| | HPLC/MS | 553 | | | Note [12], O-2060-01 |
| | GC/MS | | | | Note [11], O-1126-95 |
| 14. Carbophenothion | GC | 617 | 6630 B-2000 | | Note [4], page 27; Note [6], p. S73 |
| 15. Chlordane | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 16. Chloropropham | TLC | | | | Note [3], p. 104; Note [6], p. S64 |
| | HPLC | 632 | | | |
| 17. 2,4-D | GC | 615 | 6640 B-2001 | | Note [3], p. 115; Note [4], O-3105 -83 |
| | HPLC/MS | | | | Note [12],O-2060-01 |
| 18. 4,4'-DDD | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3105-83; Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000. | | |
| 19. 4,4'-DDE | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | Note [11], O-1126-95 |
| 20. 4,4'-DDT | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 21. Demeton-O | GC | 614, 622 | | | Note [3], p. 25; Note [6], p. S51 |
| 22. Demeton-S | GC | 614, 622 | | | Note [3], p. 25; Note [6], p. S51 |
| 23. Diazinon | GC | 507, 614, 622, 1657 | | | Note [3], p. 25; Note [4], O-3104-83; Note [6], p. S51 |
| | GC/MS | 525.2 | | | Note [11], O-1126-95 |
| 24. Dicamba | GC | 615 | | | Note [3], p. 115 |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| 25. Dichlofenthion | GC | 622.1 | | | Note [4], page 27; Note [6], p. S73 |
| 26. Dichloran | GC | 608.2, 617 | 6630 B-2000 | | Note [3], p. 7 |
| 27. Dicofol | GC | 617 | | | Note [4], O-3104-83 |
| 28. Dieldrin | GC | 608, 617 | 6630 B-2000 | D3086-90, D5812- | Note [3], p. 7; |

| | | | | | |
|---|---|---|---|---|---|
| | | | 6630 C-2000 | 96(02) | Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | Note [11], O-1126-95 |
| 29. Dioxathion | GC | 614.1, 1657 | | | Note [4], page 27; Note [6], p. S73 |
| 30. Disulfoton | GC | 507, 614, 622, 1657 | | | Note [3], p. 25; Note [6] p. S51 |
| | GC/MS | 525.2 | | | Note [11], O-1126-95 |
| 31. Diuron | TLC | | | | Note [3], p. 104; Note [6], p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | 553 | | | Note [12], O-2060-01 |
| 32. Endosulfan I | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 625 [5] | 6410 B-2000 | | Note [13], O-2002-01 |
| 33. Endosulfan II | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [8], 3M0222 |
| | GC/MS | 625 [5] | 6410 B-2000 | | Note [13], O-2002-01 |
| 34. Endosulfan Sulfate | GC | 608, 617 | 6630 C-2000 | | Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 35. Endrin | GC | 505, 508, 608, 617, 1656 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 525.1, 525.2, 625 [5] | 6410 B-2000 | | |
| 36. Endrin aldehyde | GC | 608, 617 | 6630 C-2000 | | Note [8], 3M0222 |
| | GC/MS | 625 | | | |
| 37. Ethion | GC | 614, 614.1,1657 | | | Note [4], page 27; Note [6], p. S73 |
| | GC/MS | | | | Note [13], O-2002-01 |
| 38. Fenuron | TLC | | | | Note [3], p. 104; Note [6], p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| 39. Fenuron-TCA | TLC | | | | Note [3], p. 104; Note [6], p. S64 |
| | HPLC | 632 | | | |
| 40. Heptachlor | GC | 505, 508, 608, 617, 1656 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7; Note [4], O-3104-83; Note [8], 3M0222 |
| | GC/MS | 525.1, 525.2, 625 | 6410 B-2000 | | |

| | | | | | |
|---|---|---|---|---|---|
| 41. Heptachlor epoxide | GC | 608, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7;<br>Note [4], O-3104-83;<br>Note [6], p. S73;<br>Note [8], 3M0222 |
| | GC/MS | 625 | 6410 B-2000. | | |
| 42. Isodrin | GC | 617 | 6630 B-2000<br>6630 C-2000 | | Note [4], O-3104-83;<br>Note [6], p. S73 |
| 43. Linuron | GC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | 553 | | | Note [12], O-2060-01 |
| | GC/MS | | | | Note [11], O-1126-95 |
| 44. Malathion | GC | 614, 1657 | 6630 B-2000 | | Note [3], p. 25; Note [6], p. S51 |
| | GC/MS | | | | Note [11], O-1126-95 |
| 45. Methiocarb | TLC | | | | Note [3], p. 94;<br>Note [6], p. S60 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| 46. Methoxychlor | GC | 505, 508, 608.2, 617, 1656 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7;<br>Note [4],<br>O-3104 -83;<br>Note [8], 3M0222 |
| | GC/MS | 525.1, 525.2 | | | Note [11], O-1126-95 |
| 47. Mexacarbate | TLC | | | | Note [3], p. 94;<br>Note [6], p.S60 |
| | HPLC | 632 | | | |
| 48. Mirex | GC | 617 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7;<br>Note [4], O-3104-83 |
| 49. Monuron | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| 50. Monuron-TCA | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| 51. Neburon | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| 52. Parathion methyl | GC | 614, 622, 1657 | 6630 B-2000 | | Note [3], p. 25;<br>Note [4], page 27 |
| | GC/MS | | | | Note [11], O-1126-95 |
| 53. Parathion ethyl | GC | 614 | 6630 B-2000 | | Note [3], p. 25;<br>Note [4], page 27 |

| | | | | | |
|---|---|---|---|---|---|
| | GC/MS | | | | Note [11], O-1126-95 |
| 54. PCNB | GC | 608.1, 617 | 6630 B-2000<br>6630 C-2000 | D3086-90,D5812-96(02) | Note [3], p. 7. |
| 55. Perthane | GC | 617 | | D3086-90, D5812-96(02) | Note [4], O-3104-83 |
| 56. Prometon | GC | 507, 619 | | | Note [3], p. 83;<br>Note [6], p. S68;<br>Note [9], O-3106-93 |
| | GC/MS | 525.2 | | | Note [11], O-1126-95 |
| 57. Prometryn | GC | 507, 619 | | | Note [3], p. 83;<br>Note [6], p. S68;<br>Note [9],O-3106-93 |
| | GC/MS | 525.1, 525.2 | | | Note [13], O-2002-01 |
| 58. Propazine | GC | 507, 619, 1656 | | | Note [3], p. 83;<br>Note [6], p. S68;<br>Note [9], O-3106-93 |
| | GC/MS | 525.1, 525.2. | | | |
| 59. Propham | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| 60. Propoxur | TLC | | | | Note [3], p. 94;<br>Note [6], p. S60 |
| | HPLC | 632 | | | |
| 61. Secbumeton | TLC | | | | Note [3], p. 83;<br>Note [6], p. S68 |
| | GC | 619 | | | |
| 62. Siduron | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note [12], O-2060-01 |
| 63. Simazine | GC | 505, 507, 619, 1656 | | | Note [3], p. 83;<br>Note [6], p. S68;<br>Note [9], O-3106-93 |
| | GC/MS | 525.1, 525.2 | | | Note [11], O-1126-95 |
| 64. Strobane | GC | 617 | 6630 B-2000<br>6630 C-2000 | | Note [3], p. 7 |
| 65. Swep | TLC | | | | Note [3], p. 104;<br>Note [6], p. S64 |
| | HPLC | 632 | | | |
| 66. 2,4,5-T | GC | 615 | 6640 B-2001 | | Note [3], p. 115;<br>Note [4], O-3105-83 |
| 67. 2,4,5-TP (Silvex) | GC | 615 | 6640 B-2001 | | Note [3], p. 115;<br>Note [4], O-3105-83 |

| | | | | | |
|---|---|---|---|---|---|
| 68. Terbuthylazine | GC | 619, 1656 | | | Note [3], p. 83;<br>Note [6], p. S68 |
| | GC/MS | | | | Note [15], O-2002-01 |
| 69. Toxaphene | GC | 505, 508, 608, 617, 1656 | 6630 B-2000<br>6630 C-2000 | D3086-90, D5812-96(02) | Note [3], p. 7;<br>Note [4], O-3105-83<br>Note [8], 3M0222 |
| | GC/MS | 525.1, 525.2, 625 | 6410 B-2000 | | |
| 70. Trifluralin | GC | 508, 617, 627, 1656 | 6630 B-2000 | | Note [3], p. 7;<br>Note [9], O-3106-93 |
| | GC/MS | 525.2 | | | Note [11], O-1126-95 |

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

[2] The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at 40 CFR Part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit.

[3] Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA. This EPA publication includes thin-layer chromatography (TLC) methods.

[4] Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3. 1987. USGS.

[5] The method may be extended to include α-BHC, γ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the 15th Edition of *Standard Methods for the Examination of Water and Wastewater.*1981. American Public Health Association (APHA).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 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.

[8] Organochlorine Pesticides and PCBs in Wastewater Using Empore ™ Disk. Revised October 28, 1994. 3M Corporation.

[9] Method O-3106-93 is in Open File Report 94-37, 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. 1994. USGS.

[10] EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, EPA 821-R-92-002, April

1992, U.S. EPA.

The full text of Methods 608 and 625 are provided at 40 CFR Part 136, Appendix A, Test Procedures for Analysis of Organic Pollutants.

EPA Methods 505, 507, 508, 525.1, 531.1 and 553 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, 1993, U.S. EPA.

EPA Method 525.2 is in Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, Revision 2.0, 1995, U.S. EPA.

EPA methods 1656 and 1657 are in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, 1993, U.S. EPA.

[11] Method O-1126-95 is in Open-File Report 95-181, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring. 1995. USGS.

[12] Method O-2060-01 is in Water-Resources Investigations Report 01-4134, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry. 2001. USGS.

[13] Method O-2002-01 is in Water-Resources Investigations Report 01-4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of moderate-use pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry. 2001. USGS.

[14] Method O-1121-91 is in Open-File Report 91-519, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of organonitrogen herbicides in water by solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring. 1992. USGS.

SECTION 10: NR 219.04 Table E is repealed and recreated to read:

**TABLE E**

**LIST OF APPROVED RADIOLOGICAL ANALYTICAL METHODS FOR WASTEWATER**

| | | Reference (method number or page) | | | | |
|---|---|---|---|---|---|---|
| **Parameter and units** | **Method** | **EPA [1]** | **Standard Methods 18th, 19th, 20th Ed.** | **Standard Methods Online** | **ASTM** | **USGS [2]** |
| 1. Alpha-Total, pCi per liter | Proportional or scintillation counter | 900.0 | 7110 B | 7110 B-00 | D1943-90, 96 | pp. 75 and 78 [3] |
| 2. Alpha-Counting error, pCi per liter | Proportional or scintillation counter | Appendix B | 7110 B | 7110 B-00 | D1943-90, 96 | p. 79 |
| 3. Beta-Total, pCi per liter | Proportional counter | 900.0 | 7110 B | 7110 B-00 | D1890-90, 96 | pp. 75 and 78 [3] |
| 4. Beta-Counting error, pCi | Proportional counter | Appendix B | 7110 B | 7110 B-00 | D1890-90, 96 | p. 79 |
| 5. (a) Radium Total pCi per liter | Proportional counter | 903.0 | 7500-Ra B | 7500-Ra B-01 | D2460-90, 97 | |
| (b) Radium, pCi per liter | Scintillation counter | 903.1 | 7500-Ra C | 7500-Ra C-01 | D3454-91, 97 | p. 81 |

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

[2] 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).

[3] 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 11: NR 219.04 Table EM is repealed and recreated to read:

Table EM
List of Approved Analytical Methods for Sludge

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

| | | | | | | |
|---|---|---|---|---|---|---|
| | Inductively Coupled 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 [18th, 19th, 21st], 3113 B−99 |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20th, 21st], 3120 B−99 |
| | Inductively Coupled 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 [18th, 19th, 21st], 3113 B−99 |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20th,21st], 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 [18th,19th, 21st], 3111 B−99 or C−99 |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20th, 21st], 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 | 200.9* | 3113 B [18th, 19th,21st], 3113 B−99 |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20th,21st], 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 [18th, 19th, 21st], 3111 B−99 or C−99 |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20th,21st], 3120 B−99 |
| | Inductively Coupled Plasma/ Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | |
| **Organics** | | | | | | |
| 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], Appendix H[10] |
| Fecal Coliform | Most Probable Number Membrane Filter | | | 9221 E [18th, 19th, 20th, 21st], 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 [18th, 19th, 20th, 21st], 2710 B−04 | Appendix D.2.[10] |
| Salmonella | Most Probable Number | | | | 9260 D.1[8] |
| | Selective Media Culture | | | | Appendix G[10] |
| **Physical** | | | | | |
| Solids | Gravimetric | | | 2540 G [18th, 19th, 20th, 21st], 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 Infor mation 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 12: NR 219.04 Table ES is repealed and recreated to read:

## TABLE ES—LIST OF APPROVED METHODS FOR PHARMACEUTICAL POLLUTANTS

| Pharmaceuticals pollutants | CAS registry No. | Analytical method number |
|---|---|---|
| acetonitrile | 75-05-8 | 1666/1671/D3371/D3695 |
| n-amyl acetate | 628-63-7 | 1666/D3695 |
| n-amyl alcohol | 71-41-0 | 1666/D3695 |
| Benzene | 71-43-2 | D4763/D3695/502.2/524.2 |
| n-butyl-acetate | 123-86-4 | 1666/D3695 |
| tert-butyl alcohol | 75-65-0 | 1666 |
| chlorobenzene | 108-90-7 | 502.2/524.2 |
| chloroform | 67-66-3 | 502.2/524.2/551 |
| o-dichlorobenzene | 95-50-1 | 1625C/502.2/524.2 |
| 1,2-dichloroethane | 107-06-2 | D3695/502.2/524.2 |
| diethylamine | 109-89-7 | 1666/1671 |
| dimethyl sulfoxide | 67-68-5 | 1666/1671 |
| Ethanol | 64-17-5 | 1666/1671/D3695 |
| ethyl acetate | 141-78-6 | 1666/D3695 |
| n-heptane | 142-82-5 | 1666/D3695 |
| n-hexane | 110-54-3 | 1666/D3695 |
| isobutyraldehyde | 78-84-2 | 1666/1667 |
| isopropanol | 67-63-0 | 1666/D3695 |
| isopropyl acetate | 108-21-4 | 1666/D3695 |
| isopropyl ether | 108-20-3 | 1666/D3695 |
| methanol | 67-56-1 | 1666/1671/D3695 |
| Methyl Cellosolve Δ | 109-86-4 | 1666/1671 |
| methylene chloride | 75-09-2 | 502.2/524.2 |
| methyl formate | 107-31-3 | 1666 |
| 4-methyl-2-pentanone (MIBK) | 108-10-1 | 1624C/1666/D3695/D4763/524.2 |
| Phenol | 108-95-2 | D4763 |
| n-propanol | 71-23-8 | 1666/1671/D3695 |
| 2-propanone (acetone) | 67-64-1 | D3695/D4763/524.2 |
| Tetrahydrofuran | 109-99-9 | 1666/524.2 |
| Toluene | 108-88-3 | D3695/D4763/502.2/524.2 |
| Triethlyamine | 121-44-8 | 1666/1671 |
| Xylenes | (Note 1) | 1624C/1666 |

[1] 1624C: m-xylene 108-38-3, o,p-xylene E-14095 (Not a CAS number; this is the number provided in the Environmental Monitoring Methods Index (EMMI) database.); 1666: m,p-xylene 136777-61-2, o-xylene 95-47-6

[1a] Test methods listed in Table C may be used for the parameters listed in this table.

[1m] EPA Methods 1666, 1667, and 1671 listed in the table above are published in the compendium titled Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters (EPA 821−B−98−016). EPA Methods 502.2 and 524.2 have been incorporated by reference into 40 CFR 141.24 and are in Methods for the Determination of Organic Compounds in Drinking Water, EPA−600/4−88−039, December 1988, Revised, July 1991, and Methods for the Determination of Organic Compounds in Drinking Water−Supplement II, EPA−600/R−92−129, August 1992, respectively. These EPA test method compendia are available from the National Technical Information Service, NTIS PB91−231480 and PB92−207703, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll−free number is 800−553−6847. ASTM test methods D3371, D3695, and D4763 are available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428−2959.

SECTION 13: NR 219.04 Table F is repealed and recreated to read:

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

| Parameter number/name | Container [1] | Preservation [2,3] | Maximum holding time [4] |
|---|---|---|---|
| **Table A—Bacterial Tests:** | | | |
| 1-5. Coliform, total, fecal, and *E. coli* | PA, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5] | 8 hours.[22,23] |
| 6. Fecal streptococci | PA, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5] | 8 hours.[22] |
| 7. Enterococci | PA, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5] | 8 hours.[22] |
| 8. *Salmonella* | PA, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5] | 8 hours.[22] |
| **Table A—Aquatic Toxicity Tests:** | | | |
| 9-12. 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], $H_2SO_4$ 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. Biochemical oxygen demand, carbonaceous | P, FP G | Cool, ≤6 °C [18] | 48 hours. |
| 15. Chemical oxygen demand | P, FP, G | Cool, ≤6 °C [18], $H_2SO_4$ 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 (or CATC) and free | P, FP, G | Cool, ≤6 °C [18], NaOH to pH >10 [6], reducing agent if oxidizer present | 14 days. |
| 25. Fluoride | P | None required | 28 days. |
| 27. Hardness | P, FP, G | $HNO_3$ or $H_2SO_4$ to pH <2 | 6 months. |
| 28. Hydrogen ion (pH) | P, FP, G | None required | Analyze within 15 minutes. |
| 31, 43. Kjeldahl and organic N | P, FP, G | Cool, ≤6 °C [18], $H_2SO_4$ 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], $H_2SO_4$ to pH <2 | 28 days. |
| 40. Nitrite | P, FP, G | Cool, ≤6 °C [18] | 48 hours. |
| 41. Oil and grease | G | Cool to ≤6 °C [18], HCl or $H_2SO_4$ to pH <2 | 28 days. |
| 42. Organic Carbon | P, FP, G | Cool to ≤6 °C [18], HCl, $H_2SO_4$, or $H_3PO_4$ to pH <2 | 28 days. |
| 44. Orthophosphate | P, FP, G | Cool, to ≤6 °C [18,24] | Filter within 15 minutes; Analyze within 48 hours. |
| 46. 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. |
| 48. Phenols | G | Cool, ≤6 °C [18], $H_2SO_4$ to pH <2 | 28 days. |
| 49. Phosphorous (elemental) | G | Cool, ≤6 °C [18] | 48 hours. |
| 50. Phosphorous, total | P, FP, G | Cool, ≤6 °C [18], $H_2SO_4$ to pH <2 | 28 days. |
| 53. Residue, total | P, FP, G | Cool, ≤6 °C [18] | 7 days. |
| 54. Residue, Filterable (TDS) | P, FP, G | Cool, ≤6 °C [18] | 7 days. |
| 55. Residue, Nonfilterable (TSS) | P, FP, G | Cool, ≤6 °C [18] | 7 days. |
| 56. Residue, Settleable | P, FP, G | Cool, ≤6 °C [18] | 48 hours. |
| 57. Residue, Volatile | P, FP, G | Cool, ≤6 °C [18] | 7 days. |
| 61. Silica | P or Quartz | Cool, ≤6 °C [18] | 28 days. |
| 64. Specific conductance | P, FP, G | Cool, ≤6 °C [18] | 28 days. |
| 65. Sulfate | P, FP, G | Cool, ≤6 °C [18] | 28 days. |
| 66. Sulfide | P, FP, G | Cool, ≤6 °C [18], add zinc acetate plus sodium hydroxide to pH >9 | 7 days. |
| 67. Sulfite | P, FP, G | None required | Analyze within 15 minutes. |
| 68. Surfactants | P, FP, G | Cool, ≤6 °C [18] | 48 hours. |

| | | | |
|---|---|---|---|
| 69. Temperature | P, FP, G | None required | Analyze. |
| 73. Turbidity | P, FP, G | Cool, ≤6 °C [18] | 48 hours. |
| **Table B—Metals:** [7] | | | |
| 10. Boron | P, FP, or Quartz | $HNO_3$ to pH <2 | 6 months. |
| 18. Chromium VI | P, FP, G | Cool, ≤6 °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] |
| 3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except boron, chromium VI, and mercury | P, FP, G | $HNO_3$ to pH <2, or at least 24 hours prior to analysis [19] | 6 months. |
| Table C—Organic Tests: [8] | | | |
| 3, 4. Acrolein and acrylonitrile | G, FP-lined septum | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$, pH to 4-5[10] | 14 days.[10] |
| 119. Adsorbable Organic Halides (AOX) | G | Cool, <6 °C, 0.008% $Na_2S_2O_3$$HNO_3$to pH <2 | Hold *at least* 3 days, but not more than 6 months. |
| 114-118. Alkylated phenols | G | Cool, <6 °C, $H_2SO_4$to pH <2 | 28 days until extraction, 40 days after extraction. |
| 7, 38. Benzidines [11, 12] | G, FP-lined cap | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$[5] | 7 days until extraction.[13] |
| 29, 35-37, 63-65, 107. Chlorinated hydrocarbons [11] | G, FP-lined cap | Cool, ≤6 °C [18] | 7 days until extraction, 40 days after extraction. |
| 120. Chlorinated Phenolics | | Cool, <6 °C, 0.008% $Na_2S_2O_3$, $H_2SO_4$ to pH <2 | 30 days until acetylation, 30 days after acetylation. |
| 15, 16, 21, 31, 87. Haloethers [11] | G, FP-lined cap | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$[5] | 7 days until extraction, 40 days after extraction. |
| 54, 55, 75, 79. Nitroaromatics and Isophorone [11] | G, FP-lined cap | Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction, 40 days after extraction. |
| 82-84. Nitrosamines [11, 14] | G, FP-lined cap | Cool, ≤6 °C[18], store in dark, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction, 40 days after extraction. |
| 88-94. PCBs [11] | G, FP-lined cap | Cool, ≤6 °C [18] | 1 year until extraction, 1 year after extraction. |
| 60-62, 66-72, 85, 86, 95-97, 102, 103. PCDDs/PCDFs [11] | | | |
| Aqueous Samples: Field and Lab Preservation | G | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$[5], pH <9 | 1 year. |
| Solids and Mixed-Phase Samples: Field | G | Cool, ≤6 °C [18] | 7 days. |

| | | | |
|---|---|---|---|
| Preservation | | | |
| Tissue Samples: Field Preservation | G | Cool, ≤6 °C [18] | 24 hours. |
| Solids, Mixed-Phase, and Tissue Samples: Lab Preservation | G | Freeze, ≤ −10 °C | 1 year. |
| 23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols [11] | G, FP-lined cap | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ | 7 days until extraction, 40 days after extraction. |
| 14, 17, 48, 50-52. Phthalate esters [11] | G, FP-lined cap | Cool, ≤6 °C [18] | 7 days until extraction, 40 days after extraction. |
| 1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons [11] | G, FP-lined cap | Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction, 40 days after extraction. |
| 6, 57, 106. Purgeable aromatic hydrocarbons | G, FP-lined septum | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$[5], HCl to pH 2 [9] | 14 days.[9] |
| 13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons | G, FP-lined septum | Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$[5] | 14 days. |
| **Table D—Pesticides Tests:** | | | |
| 1-70. Pesticides [11] | G, FP-lined cap | Cool, ≤6 °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. |
| **Table H—Bacterial Tests:** | | | |
| 1.*E. coli* | PA, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5] | 8 hours.[22] |
| 2. Enterococci | PA, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5] | 8 hours.[22] |
| **Table H—Protozoan Tests:** | | | |
| 8.*Cryptosporidium* | LDPE; field filtration | 1-10 °C | 96 hours.[21] |
| 9.*Giardia* | LDPE; field filtration | 1-10 °C | 96 hours.[21] |

[1] "P" is for 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 and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (e.g., using a 24-hour composite sampler, refrigerate the sample at ≤ 6 °C during collection unless specified otherwise in this table or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤ 6 °C, unless specified otherwise 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 a grab sample, a composite sample, or aliquot split from a 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 melt- water 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 requirement, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater; Nitric acid ($HNO_3$) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid ($H_2SO_4$) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. 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 EPA Regional Administrator under s. NR219.05). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler); the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See 40 CFR 136.3(e) for details.

[5] ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g.,chlorine). Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (20th and 21st editions) addresses dechlorination procedures.

[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 □g or mg), and d
cyanide con- centration.

(1) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately con- duct 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., CubitainerTM). 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 col- lapsing 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 g or mg), and di 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 sam- ple. 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 form 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. 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. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

[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. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

[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 pH < 2, then the sample must be analyzed within seven days of sampling.

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

[11] When the extractable analytes of concern fall within a single chemical category, the specified 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 ≤ 6 °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 °C.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

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

[16] Place sufficient ice 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, immediately measure the temperature of the samples and confirm that the preservation temperature maximum 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. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.

[17] Samples collected for the determination of trace level mercury (<100 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 ≤ 6 °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. Also, for purposes of NPDES monitoring, the specification of "≤ 6 °C" is used in place of the "4 °C" and "< 4 °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 ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 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] Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

[23] For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB-EC) or 1681 (A-1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

[24] The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (*i.e.,*that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (*i.e.,*within 15 minutes of collection).

SECTION 14: NR 219.04 Table G is created to read:

**TABLE G—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS**

| EPA survey code | Pesticide name | CAS No. | EPA analytical method No.(s) [3] |
|---|---|---|---|
| 8 | Triadimefon | 43121-43-3 | 1656, 507, 633, 525.1, 525.2 |
| 12 | Dichlorvos | 62-73-7 | 1657, 507, 525.1, 525.2, 622 |
| 16 | 2,4-D; 2,4-D Salts and Esters [2,4-Dichloro-phenoxyacetic acid] | 94-75-7 | 1658, 515.1, 515.2, 555, 615 |
| 17 | 2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid] | 94-82-6 | 1658, 515.1, 515.2, 555, 615 |
| 22 | Mevinphos | 7786-34-7 | 1657, 507, 525.1, 525.2, 622 |
| 25 | Cyanazine | 21725-46-2 | 629, 507 |
| 26 | Propachlor | 1918-16-7 | 1656, 508, 608.1, 525.1, 525.2 |
| 27 | MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid] | 94-74-6 | 1658, 555, 615 |
| 30 | Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid] | 120-36-5 | 1658, 515.1, 515.2, 555, 615 |
| 31 | MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid] | 93-65-2 | 1658, 555, 615 |
| 35 | TCMTB [2-(Thiocyanomethylthio) benzo-thiazole] | 21564-17-0 | 637 |
| 39 | Pronamide | 23950-58-5 | 507, 525.1, 525.2, 633.1 |
| 41 | Propanil | 709-98-8 | 1656, 632.1 |
| 45 | Metribuzin | 21087-64-9 | 1656, 507, 525.1, 525.2, 633 |
| 52 | Acephate | 30560-19-1 | 1656, 1657 |
| 53 | Acifluorfen | 50594-66-6 | 515.1, 515.2, 555 |
| 54 | Alachlor | 15972-60-8 | 1656, 505, 507, 525.1, 525.2, 645 |
| 55 | Aldicarb | 116-06-3 | 531.1 |
| 58 | Ametryn | 834-12-8 | 507, 525.2, 619 |
| 60 | Atrazine | 1912-24-9 | 1656, 505, 507, 525.1, 525.2, 619 |
| 62 | Benomyl | 17804-35-2 | 631 |
| 68 | Bromacil; Bromacil Salts and Esters | 314-40-9 | 1656, 507, 525.1, 525.2, 633 |
| 69 | Bromoxynil | 1689-84-5 | 1625, 1661 |
| 69 | Bromoxynil octanoate | 1689-99-2 | 1656 |
| 70 | Butachlor | 23184-66-9 | 1656, 507, 525.1, 525.2, 645 |
| 73 | Captafol | 2425-06-1 | 1656 |
| 75 | Carbaryl [Sevin] | 63-25-2 | 531.1, 553, 632 |
| 76 | Carbofuran | 1563-66-2 | 531.1, 632 |
| 80 | Chloroneb | 2675-77-6 | 1656, 508, 525.1, 525.2, 608.1 |
| 82 | Chlorothalonil | 1897-45-6 | 1656, 508, 525.1, 525.2, 608.2 |
| 84 | Stirofos | 961-11-5 | 1657, 507, 525.1, 525.2, 622 |
| 86 | Chlorpyrifos | 2921-88-2 | 1657, 508, 622 |
| 90 | Fenvalerate | 51630-58-1 | 1660 |
| 103 | Diazinon | 333-41-5 | 1657, 507, 525.2, 614, 622 |
| 107 | Parathion methyl | 298-00-0 | 1657, 614, 622 |

| | | | |
|---|---|---|---|
| 110 | DCPA<br>[Dimethyl 2,3,5,6-tetrachloro-terephthalate] | 1861-32-1 | 1656, 508, 525.1, 525.2, 515.1 [2], 515.2 [2], 608.2 |
| 112 | Dinoseb | 88-85-7 | 1658, 515.1, 515.2, 555, 615 |
| 113 | Dioxathion | 78-34-2 | 1657, 614.1 |
| 118 | Nabonate<br>[Disodium cyanodithioimidocarbonate] | 138-93-2 | 630.1 |
| 119 | Diuron | 330-54-1 | 553, 632 |
| 123 | Endothall | 145-73-3 | 548, 548.1 |
| 124 | Endrin | 72-20-8 | 1656, 505, 508, 525.1, 525.2, 608, 617 |
| 125 | Ethalfluralin | 55283-68-6 | 1656, 627 See footnote 1 |
| 126 | Ethion | 563-12-2 | 1657, 614, 614.1 |
| 127 | Ethoprop | 13194-48-4 | 1657, 507, 525.1, 525.2, 622 |
| 132 | Fenarimol | 60168-88-9 | 1656, 507, 525.1, 525.2, 633.1 |
| 133 | Fenthion | 55-38-9 | 1657, 622 |
| 138 | Glyphosate [N-(Phosphonomethyl) glycine] | 1071-83-6 | 547 |
| 140 | Heptachlor | 76-44-8 | 1656, 505, 508, 525.1, 525.2, 608, 617 |
| 144 | Isopropalin | 33820-53-0 | 1656, 627 |
| 148 | Linuron | 330-55-2 | 553, 632 |
| 150 | Malathion | 121-75-5 | 1657, 614 |
| 154 | Methamidophos | 10265-92-6 | 1657 |
| 156 | Methomyl | 16752-77-5 | 531.1, 632 |
| 158 | Methoxychlor | 72-43-5 | 1656, 505, 508, 525.1, 525.2, 608.2, 617 |
| 172 | Nabam | 142-59-6 | 630, 630.1 |
| 173 | Naled | 300-76-5 | 1657, 622 |
| 175 | Norflurazon | 27314-13-2 | 1656, 507, 525.1, 525.2, 645 |
| 178 | Benfluralin | 1861-40-1 | 1656, 627 See footnote 1 |
| 182 | Fensulfothion | 115-90-2 | 1657, 622 |
| 183 | Disulfoton | 298-04-4 | 1657, 507, 525,2, 614, 622 |
| 185 | Phosmet | 732-11-6 | 1657, 622.1 |
| 186 | Azinphos Methyl | 86-50-0 | 1657, 614, 622 |
| 192 | Organo-tin pesticides | 12379-54-3 | 200.7, 200.9, Ind-01 |
| 197 | Bolstar | 35400-43-2 | 1657, 622 |
| 203 | Parathion | 56-38-2 | 1657, 614 |
| 204 | Pendimethalin | 40487-42-1 | 1656 |
| 205 | Pentachloronitrobenzene | 82-68-8 | 1656, 608.1, 617 |
| 206 | Pentachlorophenol | 87-86-5 | 1625, 515.2, 555, 515.1, 525.1, 525.2, 625 |
| 208 | Permethrin | 52645-53-1 | 1656, 1660, 508, 525.1, 525.2, 608.2 |
| 212 | Phorate | 298-02-2 | 1657, 622 |
| 218 | Busan 85 [Potassium dimethyldithiocarbamate] | 128-03-0 | 630, 630.1 |
| 219 | Busan 40<br>[Potassium N-hydroxymethyl-N-methyldithiocarbamate] | 51026-28-9 | 630, 630.1 |
| 220 | KN Methyl<br>[Potassium N-methyl-dithiocarbamate] | 137-41-7 | 630, 630.1 |
| 223 | Prometon | 1610-18-0 | 507, 525.2, 619 |
| 224 | Prometryn | 7287-19-6 | 507, 525.1, 525.2, 619 |
| 226 | Propazine | 139-40-2 | 1656, 507, 525.1, 525.2, 619 |
| 230 | Pyrethrin I | 121-21-1 | 1660 |

| | | | |
|---|---|---|---|
| 232 | Pyrethrin II | 121-29-9 | 1660 |
| 236 | DEF [S,S,S-Tributyl phosphorotrithioate] | 78-48-8 | 1657 |
| 239 | Simazine | 122-34-9 | 1656, 505, 507, 525.1, 525.2, 619 |
| 241 | Carbam-S<br>[Sodium dimethyldithio-carbamate] | 128-04-1 | 630, 630.1 |
| 243 | Vapam [Sodium methyldithiocarbamate] | 137-42-8 | 630, 630.1 |
| 252 | Tebuthiuron | 34014-18-1 | 507, 525.1, 525.2 |
| 254 | Terbacil | 5902-51-2 | 1656, 507, 525.1, 525.2, 633 |
| 255 | Terbufos | 13071-79-9 | 1657, 507, 614.1, 525.1, 525.2 |
| 256 | Terbuthylazine | 5915-41-3 | 1656, 619 |
| 257 | Terbutryn | 886-50-0 | 507, 525.1, 525.2, 619 |
| 259 | Dazomet | 533-74-4 | 1659, 630, 630.1 |
| 262 | Toxaphene | 8001-35-2 | 1656, 505, 508, 525.1, 525.2, 608, 617 |
| 263 | Merphos [Tributyl phosphorotrithioate] | 150-50-5 | 1657, 507, 525.1, 525.2, 622 |
| 264 | Trifluralin [1] | 1582-09-8 | 1656, 508, 525.2, 617, 627 |
| 268 | Ziram [Zinc dimethyldithiocarbamate] | 137-30-4 | 630, 630.1 |

[1] Monitor and report as total Trifluralin.

[2] Applicable to the analysis of DCPA degradates.

[3] EPA Methods 608.1 through 645, 1645 through 1661, and Ind-01 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, Revision I, August 1993, U.S. EPA. EPA Methods 200.9 and 505 through 555 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, August 1993, U.S. EPA. The full text of Methods 608, 625 and 1625 are provided at Appendix A of this Part 136. The full text of Method 200.7 is provided at appendix C of this part 136.

SECTION 15: NR 219.04 Table H is created to read:

**TABLE H—LIST OF APPROVED MICROBIOLOGICAL METHODS FOR AMBIENT WATER**

| Parameter and units | Method [1] | EPA | Standard methods | 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 | p. 132 [3] | 9221 C E-2006 | | |
| | Membrane filter (MF) [2], single step | p. 124 [3] | 9222 D-1997 | B-0050-85 [4] | |
| 2. Coliform (fecal) in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 132 [3] | 9221 C, E-2006 | | |
| | MF [2], single step [5] | p. 124 [3] | 9222 D-1997 | | |
| 3. Coliform (total), number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 [3] | 9221 B-2006 | | |
| | MF [2], single step or two step | p. 108 [3] | 9222 B-1997 | B-0025-85 [4] | |
| 4. Coliform (total), in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 [3] | 9221 B-2006 | | |
| | MF [2] with enrichment | p. 111 [3] | 9222 (B+B.5c)-1997 | | |
| 5. *E. coli*, number per 100 mL | MPN [6, 8, 14], multiple tube, or | | 9221 B.1-2006/ 9221 F-2006 [11, 13] | | |
| | Multiple tube/multiple well, or | | 9223 B-2004 [12] | 991.15 [10] | Colilert® [12, 16] Colilert-18® [12, 15, 16] |
| | MF [2, 5, 6, 7, 8], two step, or | 1103.1 [19] | 9222 B-1997/ 9222 G-1997 [18] 9213 D-2007 | D5392-93 [9] | |
| | Single step | 1603 [20], 1604 [21], | | | mColiBlue-24® [17] |
| 6. Fecal streptococci, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 139 [3] | 9230 B-2007 | | |
| | MF [2], or | p. 136 [3] | 9230 C-2007 | B-0055-85 [4] | |
| | Plate count | p. 143 [3] | | | |
| 7. Enterococci, number per 100 mL | MPN [6, 8], multiple tube/multiple well, or | | | D6503-99 [9] | Enterolert® [12,,22] |
| | MF [2, 5, 6, 7, 8] two step, or | 1106.1 [23] | 9230 C-2007 | D5259-92 [9] | |
| | Single step, or | 1600 [24] | 9230 C-2007 | | |
| | Plate count | p. 143 [3] | | | |
| **Protozoa:** | | | | | |

| | | |
|---|---|---|
| 8.*Cryptosporidium* | Filtration/IMS/FA | 1622 [25]<br>1623 [26] |
| 9.*Giardia* | Filtration/IMS/FA | 1623 [26] |

[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] Microbiological Methods for Monitoring the Environment, Water, and Wastes. EPA/600/8-78/017. 1978. US EPA.

[4] 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. 1989. USGS.

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

[6] 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.

[7] When the MF method has not 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.

[8] 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.

[9] Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. 2000, 1999, 1996. ASTM International.

[10] Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. 1995. AOAC International.

[11] The multiple-tube fermentation test is used in 9221B.1-2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB 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.

[12] These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by*E. coli.*

[13] After prior enrichment in a presumptive medium for total coliform using 9221B.1-2006, 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-2006. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

[14] Samples shall be enumerated by the multiple-tube or multiple-well 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® or Quanti-Tray®/2000, and the MPN calculated from the table provided by the manufacturer.

[15] 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.

[16] Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories Inc.

[17] A description of the mColiBlue24® test may be obtained from Hach Company.

[18] Subject total coliform positive samples determined by 9222B-1997 or other membrane filter procedure to 9222G-1997 using NA-MUG media.

[19] Method 1103.1:*Escherichia coli*(*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant*Escherichia coli*Agar (mTEC), EPA-821-R-10-002. March 2010. US EPA.

[20] Method 1603:*Escherichia coli*(*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant*Escherichia coli*Agar (Modified mTEC), EPA-821-R-09-007. December 2009. US EPA.

[21] Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. New Medium for the Simultaneous Detection of Total Coliform and*Escherichia coli*in Water. Appl. Environ. Microbiol. 59:3534-3544 and in Method 1604: Total Coliforms and*Escherichia coli*(*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium), EPA 821-R-02-024, September 2002, US EPA.

[22] A description of the Enterolert® test may be obtained from IDEXX Laboratories Inc.

[23] Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA), EPA-821-R-09-015. December 2009. US EPA.

[24] Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA-821-R-09-016. December 2009. US EPA.

[25] Method 1622 uses a 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.*Method 1622:*Cryptosporidium*in Water by Filtration/IMS/FA, EPA-821-R-05-001. December 2005. US EPA.

[26] Method 1623 uses a 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. Method 1623.*Cryptosporidium*and*Giardia*in Water by Filtration/IMS/FA. EPA-821-R-05-002. December 2005. US EPA

SECTION 16. EFFECTIVE DATE. This rule shall take effect on the first day of the month following publication in the Wisconsin administrative register as provided in s. 227.22 (2) (intro.), Stats.

SECTION 17. BOARD ADOPTION**.** This rule was approved and adopted by the State of Wisconsin Natural Resources Board on _____.

Dated at Madison, Wisconsin _____.

STATE OF WISCONSIN
DEPARTMENT OF NATURAL RESOURCES

By _____
Cathy Stepp, Secretary

(SEAL)