ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 122, 136, 141, 143, 430, 455, and 465

[EPA–HQ–OW–2003–0070; FRL–8203–8]

RIN 2040–AD71

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This rule modifies the testing procedures approved for analysis and sampling under the Clean Water Act and Safe Drinking Water Act. EPA proposed these changes for public comment on August 18, 2003 and April 6, 2004. The Clean Water Act changes adopted in this final rule fall into the following categories: new vendor-developed methods as well as EPA and voluntary consensus standard bodies (VCSB) methods, updated versions of currently approved methods, revisions to method modification and analytical requirements, withdrawal of certain outdated methods, and changes to sample collection, preservation, and holding time requirements. This rule also changes regulations under the Safe Drinking Water Act that establish drinking water sampling and analysis procedures. The changes include approval of vendor-developed methods, new EPA and VCSB methods, updated VCSB methods, and approval of a modification to the test kit used with Syngenta Method AG–625 that restricts its use in certain circumstances. The addition of new and updated methods to the wastewater and drinking water regulations provides increased flexibility to the regulated community and laboratories in the selection of analytical methods.

ADDRESSES: EPA has established a docket for this action under Docket ID No. EPA–OW–2003–0070. All documents in the docket are listed on the *http://www.regulations.gov* web site. Although listed in the index, some information is not publicly available, e.g., CBI or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, is not placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically through *http://www.regulations.gov* or in hard copy at the HQ Water Docket Center, EPA/DC, EPA West, Room 3334, 1301 Constitution Ave., NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566–1744, and the telephone number is (202) 566–2426 for the HQ Water Docket Center.

DATES: This regulation is effective April 11, 2007. The incorporation by reference of these methods is approved by the Director of the Federal Register on April 11, 2007. For judicial review purposes, this final rule is promulgated as of 1:00 p.m. (Eastern time) on March 26, 2007 as provided at 40 CFR 23.2 and 23.7.

FOR FURTHER INFORMATION CONTACT: For information regarding the changes to wastewater regulations, contact Meghan Hessenauer, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave., NW., Washington, DC 20460, 202–566–1040 (e-mail: *hessenauer.meghan@epa.gov*). For information regarding the changes to drinking water regulations, contact Patricia Snyder Fair, Technical Support Center (MS 140), USEPA, Office of Ground Water and Drinking Water, 26 West Martin Luther King Drive, Cincinnati, OH 45268, 513–569–7937 (e-mail: *fair.pat@epa.gov*).

SUPPLEMENTARY INFORMATION:

A. Potentially Regulated Entities

1. Clean Water Act

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits with conditions designed to ensure compliance with the technology-based and water quality-based requirements of the Clean Water Act (CWA). These permits may include restrictions on the quantity of pollutants that may be discharged as well as pollutant measurement and reporting requirements. If EPA has approved test procedures for analysis of a specific pollutant, the NPDES permittee must use an approved test procedure (or an approved alternate test procedure) for the specific pollutant when measuring the required waste constituent. Similarly, if EPA has established sampling requirements, measurements taken under an NPDES permit must comply with these requirements. Therefore, entities with NPDES permits will potentially be regulated by the actions in this rulemaking. Categories and entities that may potentially be subject to the requirements of today's rule include:

Category	Examples of potentially regulated entities
State, Territorial, and Indian Tribal Governments.	States, Territories, and Tribes authorized to administer the NPDES permitting program; States, Territories, and Tribes providing certification under Clean Water Act section 401.
Industry	Facilities that must conduct monitoring to comply with NPDES permits.
Municipalities	POTWs that must conduct monitoring to comply with NPDES permits.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be regulated by this action. This table lists types of entities that EPA is now aware could potentially be regulated by this action. Other types of entities not listed in the table could also be regulated. To determine whether your facility is regulated by this action, you should carefully examine the applicability language at 40 CFR 122.1, (NPDES purpose and scope), 40 CFR 136.1 (NPDES permits and CWA), 40 CFR 403.1 (Pretreatment standards purpose and applicability). If you have questions regarding the applicability of this action to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

2. Safe Drinking Water Act

Public water systems are the regulated entities required to measure contaminants in drinking water samples. In addition, EPA Regions, as well as States, and Tribal governments with authority to administer the regulatory program for public water systems under the Safe Drinking Water Act, may also measure contaminants in water samples. When EPA establishes a maximum contaminant level (MCL) for a given drinking water contaminant, the Agency also approves standardized test procedures for analysis of the contaminant. Public water systems required to test water samples must use one of the approved test procedures. Categories and entities that may ultimately be subject to the requirements of today's rule include:

Category	Examples of potentially regulated entities	NAICS [1]
State, Local, & Tribal Governments	States, local and tribal governments that analyze water samples on behalf of public water systems required to conduct such analysis; States, local and tribal governments that themselves operate community and non-transient non-community water systems required to monitor.	924110
Industry	Private operators of community and non-transient non-community water systems required to monitor.	221310
Municipalities	Municipal operators of community and non-transient non-community water systems required to monitor.	924110

[1] North American Industry Classification System.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be regulated by this action. The table lists types of entities that EPA is now aware could potentially be regulated by this action. Other types of entities not listed in the table could also be regulated. To determine whether your facility is regulated by this action, you should carefully examine the applicability language at 40 CFR 141.2 (definition of public water system). If you have questions regarding the applicability of this action to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

B. What Process Governs Judicial Review of This Rule?

Under Section 509(b)(1) of the Clean Water Act (CWA), judicial review of today's CWA rule may be obtained by filing a petition for review in the United States Circuit Court of Appeals within 120 days from the date of promulgation of this rule. For judicial review purposes, this final rule is promulgated as of 1 p.m. (Eastern time) on March 26, 2007 as provided at 40 CFR 23.2. Under section 1448 (a) of the Safe Drinking Water Act (SDWA), judicial review of today's SDWA rule may be obtained by filing a petition for review only in the United States Court of Appeal for the District of Columbia Circuit within 45 days from the date of promulgation of this rule. For SDWA judicial review purposes, this final rule is promulgated as of 1 p.m. (Eastern time) on March 26, 2007 as provide at 40 CFR 23.7. The requirements of this regulation may also not be challenged later in civil or criminal proceedings brought by EPA.

C. Abbreviations and Acronyms Used in the Preamble and Final Rule

AOAC: AOAC-International
ASTM: ASTM International
ATP: Alternate Test Procedure
CWA: Clean Water Act
EPA: Environmental Protection Agency
FLAA: Flame Atomic Absorption Spectroscopy
ICP–AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP–MS: Inductively Coupled Plasma-Mass Spectroscopy
NPDES: National Pollutant Discharge Elimination System
NPDWR: National Primary Drinking Water Regulations
NSDWR: National Secondary Drinking Water Regulations
QC: Quality Control
SDWA: Safe Drinking Water Act
STGFAA: Stabilized Temperature Graphite Furnace Atomic Absorption Spectroscopy
USGS: United States Geological Survey
VCSB: Voluntary Consensus Standard Body

D. Table of Contents

I. Statutory Authority

A. Clean Water Act

EPA is promulgating today's rule pursuant to the authority of sections 301(a), 304(h), and 501(a) of the Clean Water Act ("CWA" or the "Act"), 33 U.S.C. 1311(a), 1314(h), 1361(a). Section 301(a) of the Act prohibits the discharge of any pollutant into navigable waters unless the discharge complies with a National Pollutant Discharge Elimination System (NPDES) permit issued under section 402 of the Act. Section 304(h) of the Act requires the Administrator of the EPA to "* * * promulgate guidelines establishing test procedures for the analysis of pollutants that shall include the factors which must be provided in any certification pursuant to [section 401 of this Act] or permit application pursuant to [section 402 of this Act].'' Section 501(a) of the Act authorizes the Administrator to''* * * prescribe such regulations as are necessary to carry out this function under [the Act].'' EPA generally has codified its test procedure regulations (including analysis and sampling requirements) for CWA programs at 40 CFR Part 136, though some requirements are codified in other Parts (e.g., 40 CFR Chapter I, Subchapters N and O).

B. Safe Drinking Water Act

The Safe Drinking Water Act (SDWA), as amended in 1996, requires EPA to promulgate national primary drinking water regulations (NPDWRs) that specify maximum contaminant levels (MCLs) or treatment techniques for drinking water contaminants (SDWA section 1412 (42 U.S.C. 300g–1)). NPDWRs apply to public water systems pursuant to SDWA sections 1401(1)(A) (42 U.S.C. 300f(1)(A)). According to SDWA section 1401(1)(D), NPDWRs include ''* * * criteria and procedures to assure a supply of drinking water which dependably complies with such maximum contaminant levels; including accepted methods for quality control and testing procedures * * *'' (42 U.S.C. 300f(1)(D)). In addition, SDWA section 1445(a) authorizes the Administrator to establish regulations for monitoring to help determine whether persons are acting in compliance with the requirements of the SDWA (42 U.S.C. 300j–4). EPA's promulgation of analytical methods for NPDWRs is authorized under these sections of the SDWA as well as the general rulemaking authority in SDWA section 1450(a) (42 U.S.C. 300j–9(a)).

The SDWA also authorizes EPA to promulgate national secondary drinking water regulations (NSDWRs) for contaminants in drinking water that primarily affect the aesthetic qualities relating to the public acceptance of drinking water (SDWA section 1412 (42 U.S.C. 300g–1)). These regulations are not Federally enforceable but are guidelines for the States (40 CFR 143.1). The NSDWRs also include analytical techniques for determining compliance with the regulations (40 CFR 143.4). EPA's promulgation of analytical methods for NSDWRs is authorized under general rulemaking authority in SDWA section 1450(a) (42 U.S.C. 300j–9(a)).

II. Summary of Final Rule

The following sections describe the changes EPA is making in today's final rule.

A. 40 CFR Part 122

1. Some EPA Effluent Guideline regulations, as well as EPA's Sewage Sludge Use and Disposal Regulations, require the use of analytical methods specifically prescribed by the Guideline (see e.g. 40 CFR Part 420.21(c)). This rule amends the National Pollutant Discharge Elimination System (NPDES) regulations at 40 CFR Part 122 to clarify when an NPDES permittee must use for its monitoring and reporting the analytical methods specifically required in Title 40 of the CFR, Chapter I, Subchapters N and O (effluent guidelines and sewage sludge, respectively) rather than the methods in 40 CFR Part 136. The regulatory language has been amended to explicitly require either use of methods at 40 CFR Part 136, or other methods that are specifically prescribed for the particular wastestream or for sewage sludge monitoring requirements.

2. The rule also corrects inconsistencies in sampling requirements by referencing back to 40 CFR Part 136 to explicitly require use of procedures at 40 CFR Part 136.

3. Sections 122.21(g)(7), 122.21(h)(4)(i), and 122.21(j)(4)(viii) require an NPDES permit applicant to report quantitative data for certain pollutants. The rule adds *E. coli* and Enterococci to the list of parameters at 40 CFR 122.21(g)(7) and 122.21(h)(4)(i), and adds *E. coli* to the list of parameters at 40 CFR 122.21(j)(4)(viii).

B. 40 CFR Part 136

This rule approves new and revised methods for inclusion in 40 CFR Part 136. Many of those methods are from the voluntary consensus standards body (VCSB) that publishes Standard Methods for the Examination of Water and Wastewater. Those ''Standard Methods'' are now available in both hard copy and electronic formats. The publisher identifies Standard Methods in two ways, depending on the medium of publication. Hard copy methods are identified by the method number (e.g., SM 4500–Cl E) and the edition of the manual in which they appear (e.g., 18th, 19th, 20th Editions), while the electronic versions available on-line are identified by the same method number and the year the method was approved for release (e.g., 4500–Cl E–00).

The following discussion briefly describes the changes to Part 136 methods approved today.

1. This rule amends the regulations at 40 CFR Part 136 to approve three new methods submitted as alternate test procedures for monitoring chemical pollutants. These methods are ''Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte'' (D6508, Rev. 2) by Waters Corporation, ''Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis'' (QuikChem Method 10–204–00–1–X) by Lachat Instruments, and ''Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate'' (Kelada–01) by Dr. Nabih Kelada.

2. The rule approves a broad purpose digestion procedure for total recoverable elements digestion. EPA is approving EPA Method 200.2 (Revision 2.8, 1994) for use with Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP–AES), Inductively Coupled Plasma-Mass Spectroscopy (ICP–MS), Stabilized Temperature Graphite Furnace Atomic Absorption (STGFAA), and Flame Atomic Absorption Spectroscopy (FLAA) methods.

3. The rule approves three new multi-element test procedures. The approved methods are EPA Method 200.8 (Revision 5.4, 1994), AOAC Method 993.14 [16th Edition], and ASTM D5673–03 for determination of elements by ICP–MS.

4. EPA is also approving a new multi-element test procedure, EPA Method 200.9 (Revision 2.2, 1994), for determination of elements by STGFAA.

5. The rule approves four new methods for determination of hexavalent chromium by ion chromatography. The methods are EPA Method 218.6, AOAC Method 993.23 [16th Edition], and ASTM D5257–97—SM 3500–Cr C [20th Edition] and SM 3500–Cr E [18th, 19th Editions].

6. The rule approves five new methods for determination of anions by ion chromatography. The methods are EPA Method 300.0 (Revision 2.1, 1993), EPA Method 300.1 (Revision 1.0, 1997), AOAC Method 993.30 [16th Edition], ASTM D4327, and SM 4110 B [18th, 19th, and 20th Editions].

7. The rule approves the use of six automated cadmium reduction methods to determine nitrate and nitrite individually, as well as in combination. The methods are EPA Method 353.2 (Revision 2.0, 1993), ASTM D3867–99 (A) and (B), SM 4500–NO_3^-E and F [18th, 19th, 20th Editions] and 4500–NO_3^-E and F (2000), and USGS Method I–4545–85.

8. The rule approves a new method for determination of chlorine by low level amperometry—SM 4500–Cl E [18th, 19th, 20th Editions] and SM 4500–Cl E (2000).

9. The rule replaces EPA Method 180.1 (1978) for determination of turbidity with EPA Method 180.1 (Revision 2.0, 1993).

10. The rule replaces EPA Method 200.7 (1990) for determination of elements by ICP–AES with EPA Method 200.7 (Revision 4.4, 1994).

11. The rule replaces EPA Method 245.1 (1974) for determination of mercury with EPA Method 245.1 (Revision 3.0, 1994).

12. The rule replaces EPA Method 335.3 (1978) for determination of total cyanide with EPA Method 335.4 (Revision 1.0, 1993) with a footnote to clarify the proper procedure for removing sulfide interferences.

13. The rule replaces EPA Method 350.1 (1978) for determination of ammonia with EPA Method 350.1 (Revision 2.0, 1993).

14. The rule replaces EPA Method 351.2 (1978) for determination of total Kjeldahl nitrogen (TKN) with EPA Method 351.2 (Revision 2.0 1993).

15. The rule replaces EPA Method 353.2 (1978) for determination of nitrate-nitrite with EPA Method 353.2 (Revision 2.0, 1993).

16. The rule replaces EPA Method 365.1 (1978) for determination of phosphorus (all forms) with EPA Method 365.1 (Revision 2.0, 1993).

17. The rule replaces EPA Method 375.2 (1978) for determination of sulfate with EPA Method 375.2 (Revision 2.0, 1993).

18. The rule replaces EPA Method 410.4 (1978) for determination of chemical oxygen demand (COD) with EPA Method 410.4 (Revision 2.0, 1993).

19. The rule replaces EPA Method 420.2 (1974) for determination of total phenols with EPA Method 420.4 (Revision 1.0, 1993).

20. The rule approves a new method for the determination of mercury, EPA Method 245.7 ''Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry'' [Revision 2.0, 2005] (EPA–821–R–05–001).

21. The rule approves a new method for determination of available cyanide by ligand exchange followed by flow injection analysis, ASTM D6888–04.

22. The rule approves a new method for determination of cations by ion chromatography, ASTM D6919–03.

23. The rule approves a new method for determination of chloride by potentiometry, SM 4500–Cl$^-$D [18th, 19th, 20th Editions] and SM 4500–Cl$^-$D (2000).

24. The rule approves a new method for determination of chloride by ion selective electrode, ASTM D512–89 (1999).

25. The rule approves two new methods for determination of total cyanide by ion selective electrode, SM 4500–CN$^-$F [18th, 19th, 20th Editions] and SM 4500–CN$^-$F (2000), and ASTM D2036–98 A.

26. The rule approves two new methods for determination of sulfide by ion selective electrode, SM 4500–S_2^-G [18th, 19th, 20th Editions] and ASTM D4658–03 (1996).

27. The rule approves a new method for determination of nitrate by ion selective electrode, SM 4500–NO_3^-[18th, 19th, 20th Editions] and SM 4500–NO_3^-(2000).

28. The rule approves an errata sheet to correct typographical errors in the following methods manuals, ''Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms,'' Fourth Edition, U.S. Environmental Protection Agency, Office of Water, Washington DC, EPA/821/R–02/013 (the ''freshwater chronic manual''), and ''Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms,'' Fifth Edition, U.S. Environmental Protection Agency, Office of Water, Washington DC, EPA/821/R–02/012 (the ''freshwater acute manual'').

29. The rule approves the use of newer versions of 74 methods published by ASTM International. The new versions are published in the 1994, 1996, and 1999 Annual Book of ASTM Standards Vols. 11.01 and 11.02, in the 2000 Annual Book of ASTM Standards, Vol. 11.02 and in individual standards published after 2000.

30. The rule approves the use of newer versions of 88 methods published by the Standard Methods Committee and adopts a new numbering system to track the approved versions of Standard Methods. The new versions are published in Standard Methods Online (APHA 2003).

31. The rule approves the use of newer versions of 19 methods published by AOAC–International. The new versions of these methods are published in Official Methods of Analysis of AOAC–International, 16th Edition, 1995.

32. The rule approves the replacement of the mercuric sulfate catalyst with copper sulfate in methods approved for the determination of total Kjeldahl nitrogen (TKN).

33. The rule approves the use of styrene divinyl benzene beads and stabilized formazin as alternatives to the presently approved formazin standard for determination of turbidity.

34. As described in the preamble to the April 2004 proposed rule (69 FR 18213), EPA is adopting a new § 136.6 to introduce greater flexibility in the use of approved methods. The section describes the circumstances in which approved methods may be modified and the requirements that analysts must meet to use these modified methods in required measurements without prior EPA approval. The rule also includes language at § 136.6(c) to clarify that analysts need only meet method performance requirements for target analytes (those analytes being measured for NPDES reporting) when using multi-analyte methods for compliance monitoring purposes. The rule also includes the language at § 136.6(d) to allow explicitly the use of capillary (open tubular) GC columns with EPA Methods 601–613, 624, 625, and 1624B as alternatives to the packed GC columns specified in those methods, provided that analysts generate new retention time tables with capillary columns to be kept on file with other information for review by auditors.

35. The rule withdraws 109 methods contained in EPA's ''Methods for the Chemical Analysis of Water and Wastes'' for which approved alternatives published by voluntary consensus standards bodies (*e.g.*, ASTM and Standard Methods) are available.

36. The rule withdraws liquid-liquid extraction (LLE) methods, including EPA Methods 612 and 625, as approved procedures for determination of 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene.

37. The rule withdraws approval of all oil and grease methods that use chlorofluorocarbon-113 (CFC–113; Freon–113) as an extraction solvent.

38. The rule revises Table II (Required Containers, Preservation Techniques, and Holding Times) and the footnotes to the table at 40 CFR 136.3(e). The table and footnotes specify approved sampling, preservation, and holding time requirements for the methods approved for compliance monitoring to reduce confusion, resolve any conflicts with instructions in the underlying compliance monitoring method, and reflect current understanding of sample preservation requirements. The most significant of the changes are those made to Footnote 6, which addresses the preservation of samples to be analyzed for cyanide. Based on information gathered during the development of new cyanide methods approved in this rulemaking, and information collated from various commenters and experts in cyanide analyses, EPA revised footnote 6 to Table II by adding text that describes procedures that are recommended for removal or suppression of cyanide interferences, including interferences

from sulfur, sulfide, sulfite, thiocyanate, and aldehydes. The recommended procedures may differ from those described in the older approved methods.

39. EPA is changing ''Director of the Environmental Monitoring Systems Laboratory'' and ''Director, Analytical Methods Staff'' to ''Alternate Test Procedure Program Coordinator, Washington, DC'' every place the phrases appear in the regulations. This reflects the current organizational structure and title for the head of EPA's Alternate Test Procedure (ATP) Program management. In addition, addresses for submission of ATPs are being updated to reflect the current location of the Alternate Test Procedure Program Coordinator.

40. The rule makes other minor editorial revisions to clarify existing regulations.

C. 40 CFR Parts 141 and 143

1. This rule amends the regulations at 40 CFR Part 141 and Part 143 to allow the use of 66 methods in ''Standard Methods Online'' (APHA 2003) (40 CFR 141.21, 141.23, 141.74, and 143.4).

2. This rule allows the use of 28 newer versions of methods published by ASTM International. The new versions are published in the 1999 ''Annual Book of ASTM Standards,''' Vols.11.01 and 11.02, in the 2000 ''Annual Book of ASTM Standards,'' Vol. 11.02 and in individual standards published after 2000 (40 CFR 141.23).

3. This rule approves a new method submitted as an alternate test procedure for the determination of common anions—chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate, ''Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte'' (D6508, Rev. 2) by Waters Corporation (40 CFR 141.23 and 143.4).

4. This rule approves two new methods for determination of available cyanide, ''Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry,'' Method OIA–1677, DW (January 2004), and ASTM D6888–04 (40 CFR 141.23).

5. This rule approves the use of EPA Method 300.1 (Revision 1.0, 1997) for compliance determinations of chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate (40 CFR 141.23 and § 143.4).

6. This rule approves the use of EPA Method 552.3 (Revision 1.0, 2003) for compliance determinations of dalapon (40 CFR 141.24).

7. This rule amends 40 CFR 141.25 to add a new method for determination of radium-226 and radium-228. This method, ''The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors'' (Revision 1.2, December 2004), was developed by the Environmental Resources Center at the Georgia Institute of Technology, and was originally submitted to EPA as an alternate test procedure to the currently approved methods for determination of radium-226 and radium-228.

8. This rule allows States the option of approving ITS Free chlorine test strips as a test kit for the measurement of free chlorine using ''Free Chlorine Species'' (HOCl- and OCl-) by Test Strip,'' ITS Method D99–003 (Revision 3.0, November 21, 2003) by Industrial Test Systems, Inc. (40 CFR 141.74).

9. This rule approves EPA Method 327.0 (Revision 1.1, 2005) for measurement of chlorine dioxide residuals (40 CFR 141.74).

10. This rule approves the use of styrene divinyl benzene beads and stabilized formazin as alternatives to the presently approved formazin standard for determination of turbidity (40 CFR 141.74).

11. This rule revises footnote 17 to the table at 40 CFR 141.23 to allow the use of a 450–W UV lamp in the Kelada Method-01 for determination of cyanide.

12. This rule allows the use of Syngenta Method AG–625, with the modified immunoassay testing product manufactured by Beacon Analytical Systems, for the measurement of atrazine under certain conditions. It may only be used by those systems that do not use chlorine dioxide for drinking water treatment. In addition, the results of the analysis of samples with concentrations of atrazine more than one-half the atrazine MCL (*i.e.*, more than 1.5 µg/L) must be confirmed using another approved method (40 CFR 141.24).

13. This rules also revises footnote 8 to the table in paragraph (a)(1) to correct a long-standing discrepancy between the footnote and the specifications in Standard Method 9221 E. The table in question lists Standard Method 9221 E as one of two procedures that may be used for monitoring fecal coliforms. Footnote 8 simply notes the holding time for the A–1 broth used in this procedure. Today's action corrects the holding time specified in the footnote 8 to match the 7-day holding time that is specified in Standard Method 9221 E (40 CFR 141.74)

D. 40 CFR Part 430

This rule amends the Effluent Limitations Guidelines for the pulp, paper, and paperboard point source category at 40 CFR Part 430 to approve a new method for determination of chlorinated phenolics in wastewaters generated by these industries. The rule adds a new section, 430.02(g), to allow the use of ''Chlorinated Phenolics in Water by In situ Acetylation and GC/MS Determination'' (Method CP–86.07) developed by the National Council for Air and Stream Improvement (NCASI) as an alternative to otherwise required Method 1653 in Part 430, Appendix A.

E. 40 CFR Part 455

This rule amends the regulations at 40 CFR Part 455 by moving Table 7 from 40 CFR Part 455 to 40 CFR Part 136.3(a) as new Table IG.

F. 40 CFR Part 465

This rule amends the Effluent Limitations Guidelines for the coil coating point source category at 40 CFR Part 465 to replace the method listed at section 465.03(c) for determination of oil and grease in wastewater samples from all subcategories of coil coating with EPA Method 1664A for determination of non-polar materials (NPM), which is generally equivalent to total petroleum hydrocarbons.

III. Changes Between the Proposed Rule and the Final Rule

Except as noted below, the content of the final rule is the same as that of the proposed rule. In some instances, EPA revised for clarity the language of the final rule from that in the proposed rule.

A. Silver Determinations

EPA received comments on the Agency's proposed withdrawal of EPA Method 272.1 which included information on how to keep silver in solution in samples with known or suspected high levels of silver by adding a solution containing cyanogen chloride. As a result, EPA has added a footnote to Table IB at § 136.3 to include procedures for preparation and addition of this reagent to digested samples of this type to keep the silver in solution for analysis by any of the approved methods. The Agency is withdrawing EPA Method 272.1, as proposed in April 2004 (69 FR 18183 April 6, 2004).

B. ASTM Method D5673–02 ''Standard Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry''

Based on comment received on the Agency's proposed approval of ASTM Method D5673–02, EPA is approving an updated version of this method ASTM D5673–03. EPA included the updated version of this method in a notice of data availability (70 FR 7909, February 16, 2005) and requested public comment on whether it should be approved. EPA received no adverse comments.

C. Ammonia Determinations

EPA received comment on the Agency's proposed approval of an updated version of EPA Method 350.1. The commenter stated that mandating the use of ethylene-diamine-tetra-acetate (EDTA) in the method could adversely affect method performance when using an automated analyzer and submitted data to support this claim. Based on EPA's review of the data, the Agency is adding a footnote to Table IB at § 136.3 allowing analysts to omit EDTA from EPA Method 350.1, provided that all of the quality control (QC) acceptance criteria in the method are met.

D. Available Cyanide Determinations

The Agency solicited comment regarding problems encountered with the use of ligand exchange-amperometric detection methods for determination of available cyanide in samples that contain significant amounts of solids and solicited potential solutions to these problems. Based on public comment, EPA is adding a footnote to Table IB at § 136.3 that includes suggestions for sample filtration and for limiting the time between addition of ligand exchange reagents and analysis to solve these problems when necessary. The Agency proposed ASTM Method D6888–03 for determining available cyanide. Since publication of the proposal, EPA has received a revised version of this method. EPA included this revised version in a Notice of Data Availability (NODA) and requested public comment on the approval of this revised version of the method (70 FR 7909, February 16, 2005). D6888–04 contains a new online sulfide removal procedure and represents a refinement to the proposed version. EPA received no adverse comments on this revised version of the method and is approving ASTM Method D6888–04 in today's rule.

E. Kelada-01 Method for Determination of Available Cyanide and Total Cyanide

Based on a comment and data received on the Agency's proposed approval of the Kelada-01 method for determination of available cyanide and total cyanide for use in NPDES compliance monitoring, a note has been added at 40 CFR 136.3, Table IB, Footnote 55 to allow the use of 450–W UV lamp instead of the 550–W lamp specified in the method if it provides performance within the quality control 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. The Agency also is adding the note at 40 CFR 141.23, Footnote 17, as this method was previously approved for use in drinking water compliance monitoring.

F. Mandatory Replacement of Mercuric Sulfate With Copper Sulfate in Methods for Determination of Total Kjeldahl Nitrogen

The Agency proposed the mandatory replacement of mercury sulfate with copper sulfate in total Kjeldahl nitrogen (TKN) digestion. Comments expressed concern that copper sulfate will provide inferior performance in some samples, and suggested that EPA allow the continued use of mercury sulfate in analyses, offering copper sulfate as an option. Based on EPA's evaluation of these comments, the Agency has decided that it will continue to allow the use of mercury sulfate in TKN analyses.

G. Approval of Additional Standards for Turbidity Determinations

Based on comments received on the proposed approval of additional standards for use in methods to determine turbidity, the Agency is replacing turbidity standard trade names with more generic listings in the footnote to Table IB at § 136.3. An example formulation for each generic listing is also included in order to provide a reference to at least one source of the generic standard material. The Agency also is adding a similar footnote to the table in 40 CFR 141.74 where the approved methods for the determination of turbidity in drinking water are listed.

H. Allowed Use of Capillary Columns in EPA Methods 601–613, 624, 625, and 1624B

The Agency proposed new language that explicitly allows the use of capillary columns in place of packed columns with EPA Methods 601–613, 624, 625, and 1624B, provided that all QC tests in these methods are performed and all QC acceptance criteria are met. Based on comments received on this proposal, EPA is clarifying the regulations at § 136.6 to state that retention times will change with the switch from packed to capillary columns, so analysts are not required to meet the retention times specified in the method when the switch is made. Instead, analysts must generate new retention time tables with capillary columns to be kept on file with other information for review by auditors.

I. Changes to Sampling Requirements at 40 CFR Parts 122, 136, and 403

The Agency proposed changes to the sampling requirements specified at 40 CFR Parts 122, 136 and 403. Based on comments received on these proposed updates, the sampling requirements have been modified to include clarifying language with respect to grab and composite sampling requirements, and to add *E. coli* and Enterococci to the parameters listed at Part 122.21 for which an NPDES applicant must provide analytical information. In addition, on October 14, 2005, EPA published in the **Federal Register** final changes to its General Pretreatment Regulations (70 FR 60134). In these regulations, EPA consolidated monitoring requirements for indirect dischargers into 40 CFR 403.12(g). In the proposal for the current rule, EPA proposed to modify 40 CFR 403.12 (b). Based on changes made to 40 CFR Part 403 on October 14, 2005, however, the proposed modifications to CFR 403.12 (b) are no longer required.

J. Approval Status of Total Elements Digestion

The Agency proposed to approve a ''total recoverable'' elements digestion procedure, and also to retain ''total'' elements digestion for graphite furnace atomic absorption (GFAA) analyses. Based on comments received on this proposal, EPA is clarifying the language in Table IB at § 136.3 to specify Section 4.1.3 of Methods for Chemical Analysis of Water and Wastes (MCAWW) as the approved digestion procedure for use with non-platform graphite furnace atomic absorption determinations.

K. EPA Method 245.7 for Determination of Mercury

The Agency proposed approval of EPA Method 245.7 (December 2003) as an additional method for determination of mercury. Based on comments received on this proposal, EPA has revised the method and is approving EPA Method 245.7 (Revision 2.0, 2005) in this rule. Specifically, EPA has approved the method with the following changes: includes a discussion of the gas-liquid separator in Section 2.6, reduces the amount of hydrochloric acid used during sample digestion, includes the addition of hydrochloric acid (HCl) to standard solutions, clarifies that additional procedures for handling difficult matrices may be used provided that laboratories meet the method performance criteria, and revises procedures for preparing reagent blanks to remove the addition of stannous chloride ($SnCl_2$) and include addition of

HCl. EPA also has corrected section numbering and references throughout Sections 4 and 7 of the method.

L. Clarification of Reporting Requirements

The Agency proposed to add a new § 136.7 that would clarify that a quality control (QC) failure does not relieve a permittee of the duty to report results and that results are to be reported to the level specified in the method or required in the permit, whichever is lower. EPA agrees with public comment that the proposed revision was unclear. In this rule, EPA is revising this action to clarify our intent in text at § 136.3, rather than take final action on the proposed text at § 136.7.

EPA proposed a new § 136.7 amendment to resolve conflicts between current reporting requirements at 40 CFR Parts 122 and 125 and preservation requirements at 40 CFR 136.3(e) and instructions in compliance monitoring methods specified in the tables at 40 CFR 136.3(a). For example, some methods provide ''reject tests'' (e.g. Method 5210 B 5-day BOD, section 6a, Standard Methods, 20th edition) or state that a ''result may not be reported for regulatory compliance purposes'' (e.g. Method OIA 1677: Available Cyanide, section 9.3.4, OI Analytical) if an analytical quality control measure is outside its control limit range (acceptance criteria). However, it may be necessary to report such results if resampling, reanalysis or other corrective action is not possible. Not reporting these results may cause a permittee to be in violation of Part 122 or 125 reporting requirements. Reporting such available data is recommended as a good laboratory practice (D3856–95 Standard Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water, *Annual Book of ASTM Standards,* Water and Environmental Technology, Vol 11.01 Water (I), p. 353, 2003).

For clarification, in this rule, EPA is adding a statement to the text preceding Tables IA to IG at § 136.3(a), and Table II at § 136.3(e) to state that reporting requirements of Parts 122 or 125 may take precedence over the otherwise applicable reporting or preservation requirements of a particular analytical method.

M. Corrections to 40 CFR Part 136, Tables IA, IB, IC, ID, IE and Footnotes

The Agency is correcting errors that were published in the proposed Table IA (List of Approved Biological Methods), Table IB (List of Approved Inorganic Test Procedures), Table IC (List of Approved Test Procedures for Non-Pesticide Organic Compounds), Table ID (List of Approved Test Procedures for Pesticides), and Table IE (List of Approved Radiologic Test Procedures) and the footnotes to those tables.

N. Revisions to 40 CFR Part 136, Table II

The rule revises Table II (Required Containers, Preservation Techniques, and Holding Times), and the footnotes to Table II at 40 CFR 136.3(e). The action of updating Table II at § 136.3(e) reflects EPA's evaluation of comments received on the April 6, 2004 proposal. EPA revised footnote 1 to more clearly distinguish between polyethylene (P), fluoropolymer (FP), glass (G), and low-density polyethylene (LDPE) and has made it explicit that a sample to be used to determine fluoride is to be collected in a polyethylene bottle. EPA revised footnotes 2 and 7 to add language to make more clear that preservation must be within 15 minutes after collection of a grab sample, a composite sample, or an aliquot split from a composite sample collected automatically over time.

EPA modified footnote 4 to clarify the start of holding times for the different types of sample collection (grab samples, composite samples collected automatically, and a set of grab samples that is composited in the laboratory). The revisions remove the requirement for collection of samples to determine cyanide in amber glass bottles with PTFE-lined caps from footnote 6. Footnotes 5 and 6 describe procedures recommended for removal or suppression of cyanide interferences, including interferences from oxidants, sulfide, sulfite, thiocyanate, and aldehydes. In addition, the footnote also provides that other procedures for removal or suppression of cyanide interferences may be employed, provided the analyst demonstrates that such other procedures more accurately measure cyanide.

EPA clarified requirements in footnote 17 specific to sampling for trace mercury, added EPA Method 245.7 to footnote 17, and removed footnote 17 from the ''Preservation'' and ''Maximum Holding Time'' headers. EPA has clarified footnote 18 to indicate that the number of significant figures is intended to establish an absolute upper limit on sample temperature and preclude meeting the specification through rounding of numbers and added an exception to footnote 18 for a sample analyzed immediately (in less than 15 minutes from collection). EPA deleted footnote 19 because the information is now in footnote 5. Deletion of this footnote resulted in renumbering all of the subsequent footnotes to Table II.

EPA has also clarified that preservation is not required for soil and sediment samples. EPA also has revised preservation requirements for acrolein, acrylonitrile, and purgeable aromatics to remove inconsistences, revised requirements for collection of samples to be tested for Cryptosporidium and Giardia, and established 0–8 °C as the holding temperature for samples to be tested for Cryptosporidium. In addition, the Agency is correcting minor formatting and typographical errors that were published in the proposed Table II.

EPA recognizes that the requirements at § 136.3 may differ from those described in some older approved methods and today's action clarifies that this section supersedes instructions in a compliance method. Where there are conflicts with existing methods published by EPA, VCSBs, or other entities, we expect that the next revisions of these methods will conform to the requirements at § 136.3(e).

O. Corrections to Tables 40 CFR Parts 141.23, 141.24, 141.25, and 141.74 and Footnotes

The final rule corrects a number of errors in the proposed tables at § 141.23 (Inorganic Chemical Sampling and Analytical Requirements), 141.24 (Organic Chemicals, Sampling and Analytical Requirements), 141.25 (Analytical Methods for Radioactivity) and 141.74 (Analytical and Monitoring Requirements) and the footnotes to those tables. The Agency also revised the wording of several entries for the cyanide methods in the tables at 40 CFR 141.23 to more clearly distinguish among the various methodologies listed in those tables.

P. Retention of Syngenta Method AG–625 for Determination of Atrazine

The Agency proposed to withdraw approval of Syngenta Method AG–625 for the determination of atrazine in drinking water. Based on comments received regarding the April 6, 2004 proposal and comments received regarding additional data submitted in response to the Notice of Data Availability (70 FR 7909, February 16, 2005), EPA has decided to retain Syngenta Method AG–625 with some restrictions on its use. The revised approval requires confirmation of any compliance monitoring results for atrazine that are greater than one half of the MCL for atrazine, using an alternate approved method. EPA based its approval on the use of the modified immunoassay testing product produced by Beacon Analytical Systems, the only version of the product that is currently offered for sale. However, use is not authorized for public water systems which use chlorine dioxide for drinking water treatment.

Q. EPA Method 327.0 for Determination of Chlorine Dioxide

The Agency proposed to approve EPA Method 327.0 (Revision 1.0, 2003) for the determination of chlorine dioxide (40 CFR 141.74). In the final rule, the Agency has approved EPA Method 327.0 (Revision 1.1, 2005) and corrected three typographical errors in the proposed method.

R. Deferral of Action on Microtox® 1010

Because EPA is continuing to evaluate public comment about use of the Microtox® 1010 test procedure for determinations of acute whole effluent toxicity, and the data supporting use of this test procedure, EPA is not taking final action on the proposal to approve Microtox® 1010 for compliance monitoring.

IV. Response to Comments

EPA received two comments regarding methods included in this final rule from the August 18, 2003 proposal (68 FR 49548), 115 comments on the April 6, 2004 proposal (69 FR 18166), and nine comments on the February 16, 2005 Notice of Data Availability (NODA) (70 FR 7909). Commenters represented a number of different interests, including analytical laboratories, water utilities, instrument manufacturers, State and local governments, trade associations, scientists, and private citizens. A summary of major public comments on the proposed rules and the NODA and the Agency's responses is presented in this section, and section III of this preamble. The public docket for this rule includes the Agency's response to all comments.

A. Use of Syngenta Method AG–625 for Determination of Atrazine

EPA proposed withdrawing approval of Syngenta Method AG–625 (Syngenta Crop Protection Inc.) for the determination of atrazine. Based on comments and additional data submitted in response to the NODA, EPA has decided to retain Syngenta Method AG–625 as an approved method for atrazine, subject to certain conditions. Today's amended rule will require the use of an alternate approved method for atrazine to confirm any results from the Syngenta method that are greater than one half of the MCL for atrazine. EPA's decision to retain the approval of Method AG–625 is based on using the modified immunoassay testing product manufactured by Beacon Analytical Systems (Atrazine Plate Kit–Cat# CPP–004), the only version of the product currently offered for sale. However, based on data submitted in response to the NODA, EPA is not approving Method AG–625 for use when chlorine dioxide is used for drinking water treatment.

One commenter supported the withdrawal of Syngenta Method AG–625, citing EPA's Environmental Technology Verification (ETV) study, and suggested that the method had not been validated adequately prior to EPA's earlier approval. The commenter also suggested that approval of a modified version of AG–625 may be preferable, but was concerned that the method using the original testing product could lead to reports of non-compliance and/or increased treatment cost based on erroneous atrazine measurements in the interim.

Other comments questioned the validity of data submitted by the American Water Works Association (AWWA) that demonstrated method deficiencies and requested that the data not be considered by EPA. The commenters noted that data were not accompanied by suitable quality control checks and that some tests (*i.e.*, testing the effect of humic acid) were not reproducible in later studies by the same investigator. The commenters also suggested that data generated by AWWA was not generated according to Method AG–625, but rather by simply using the commercial testing product without reference to the method.

As reported by Adams (Adams *et al.*, JAWWA, 2004, pp. 126–139), the original immunoassay test kit used with Method AG–625 was subject to a positive bias in the measurement of atrazine in the presence of disinfectants used in drinking water treatment, such as chlorine, monochloroamine, chloramine T, chlorine dioxide and other substances. Additionally, data reported by Adams using the modified Beacon test kit, data from the Environmental Technology Verification (ETV) Program, and, in fact, data in the December 2004 Syngenta study (Atrazine: Final Report, Syngenta Number T006141–04, 12/10/2004), demonstrate that test kits marketed by Beacon, Abraxis, and SDI show a modest positive bias in raw and finished drinking water for most samples (*i.e.*, the measured concentrations in most samples were higher than the expected values). For a distinct minority of samples in these two studies, no bias or a negative bias was shown (*i.e.*, the measured concentrations were equal to or lower than the expected concentrations in a few samples). The December 2004 Syngenta study submitted to EPA demonstrated that the modified Beacon immunoassay test kit performed suitably in the presence of chlorine, monochloroamine, chloramine T, chloroform, and humic acid. However the data show that an interference occurred in the presence of chlorine dioxide unless samples were analyzed within one day of collection.

EPA agrees that data from both Syngenta and AWWA (Adams) indicate that, except for cases involving chlorine dioxide, the modified testing product performed substantially better with regard to the positive bias and interferences than did the original product in the presence of disinfectants and other interferences. EPA also agrees that, in general, the immunoassay products examined in these studies exhibit some positive bias for atrazine. Low results were only obtained from analyses of a few samples.

EPA recognizes that a potential positive bias is a concern for water utilities, particularly those located in areas such as the Midwest, where atrazine is used extensively to control weeds in corn and sorghum crops. However, in many other areas, where it is not used, atrazine is not likely to be found in drinking water samples at all. Because accurate information on the occurrence of contaminants at concentrations close to the MCL is necessary for EPA to meet its obligation to review MCLs every six years, EPA has retained approval of Syngenta Method AG–625 for atrazine with the conditions on its use described below. The following conditions have been added as Footnote 5 to the table at 40 CFR 141.24(e)(1).

> ''This method may not be used for the analysis of atrazine in any system where chlorine dioxide is used for drinking water treatment. In samples from all other systems, any result for atrazine generated by Method AG–625 that is greater than one-half the maximum contaminant level (MCL) (i.e., greater than 0.0015 mg/L or 1.5 µg/L) must be confirmed using another approved method for this contaminant and should use additional volume of the original sample collected for compliance monitoring. In instances where a result from Method AG–625 triggers such confirmatory testing, the confirmatory result is to be used to determine compliance.''

The MCL for atrazine is 0.003 mg/L (3 µg/L). Thus, any results from Method AG–625 greater than 0.0015 mg/L (1.5 µg/L) must be confirmed through the use of another approved method for atrazine. In such instances, the confirmatory result is used to determine compliance, because the methods used for confirmatory testing are considered more accurate.

EPA acknowledges that this requirement for confirmation may cause some utilities to choose not to employ Method AG–625. However, the approval of a method at 40 CFR Part 141 does not constitute a requirement for its use when other approved methods are available. Furthermore, EPA has concluded that, given the distribution of atrazine use nationwide, the majority of water utilities will not be negatively affected by the confirmation requirement and can take advantage of the potential cost savings afforded by Method AG–625 relative to other approved methods for atrazine. EPA notes that if utilities are monitoring for a broad range of contaminants using approved methods that cover multiple analytes (e.g., Method 525.2), then the use of a test kit may not offer a cost advantage because of the ability to include atrazine in a broad spectrum method.

Based on the data provided in response to the proposed rule and NODA, EPA agrees that the presence of chlorine dioxide has substantial potential to interfere with Method AG–625. Therefore, EPA has not approved the method for use in water systems where chlorine dioxide is used for drinking water treatment. While the data from one study suggest that the atrazine results are not affected if samples are analyzed within 24 hours after sample collection, the circumstances surrounding this observation in the study have not been fully characterized. At a future date, EPA may reconsider the use of Method AG–625 by water systems using chlorine dioxide, if more information is available characterizing the performance of the method.

B. Results From Use of Revised Methods, ''The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors.'' (Revision 1.2, December 2004)

The final rule establishing radionuclide drinking water standards published on December 7, 2000 (65 FR 76708) required drinking water systems to sample and report on radionuclides in their drinking water supplies during the period from December 8, 2003 to December 31, 2007, including the combined result for radium-226 and radium-228. Prior to today's rule, separate methods were required for the measurement of radium-226 and radium-228. The separate results of the two isotopes are summed to provide the ''combined'' result that is compared to the MCL. One commenter supported the approval of the method from the Georgia Institute of Technology because one method can be used to measure both radium isotopes and would make for a faster result. The commenter also noted that there are approximately 150,000 Public Water Supply source water taps across the country that would have to be sampled. Given these monitoring requirements, the commenter was concerned about sufficient laboratory capacity to meet the monitoring requirements of the December 2000 rule because approval of the Georgia Institute of Technology method would not become effective until promulgation of today's rule.

In response to this comment, EPA is allowing community water systems to use samples collected after January 1, 2005 and analyzed using the analytical method approved in this rule to satisfy the radium-226 and radium-228 monitoring requirements for the 2005–2007 compliance period. (Minor corrections to the method were made in December 2004 and are reflected in the version approved in this rule, so it was not available for analyzing samples collected prior to 2005.) Allowing this early use of the method should alleviate some of the laboratory capacity concerns. EPA has included a footnote in the revised table of analytical methods for radioactivity at § 141.25 to indicate that samples analyzed using the newly approved method are acceptable. Such ''grandfathered'' data must be based on results from the analytical method approved for use by this final rule. The term ''grandfathered data'' used in this rule does not apply to data collected to meet other grandfathering provisions specified in the radionuclide final rule that was published on December 7, 2000 (65 FR 76708).

V. Statutory and Executive Order Reviews

A. Executive Order 12866: Regulatory Planning and Review

Under Executive Order 12866 (58 FR 51735, October 4, 1993), the Agency must determine whether the regulatory action is ''significant'' and therefore subject to OMB review and the requirements of the Executive Order. The Executive Order defines ''significant regulatory action'' as one that is likely to result in a rule that may:

(1) Have an annual effect on the economy of $100 million or more, or adversely affect in a material way the economy, a sector of the economy, productivity, competition, jobs, the environment, public health or safety, or State, local, or Tribal governments or communities;

(2) Create a serious inconsistency or otherwise interfere with an action taken or planned by another agency;

(3) Materially alter the budgetary impact of entitlements, grants, user fees, or loan programs or the rights and obligations of recipients thereof; or

(4) Raise novel legal or policy issues arising out of legal mandates, the President's priorities, or the principles set forth in the Executive Order.

It has been determined that this rule is not a ''significant regulatory action'' under the terms of Executive Order 12866 and is therefore not subject to OMB review.

B. Paperwork Reduction Act

This action does not impose an information collection burden under the provisions of the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.* This rule does not impose any information collection, reporting, or recordkeeping requirements. This rule merely adds new and updated versions of testing procedures, withdraws some older testing procedures, and establishes new sample collection, preservation, and holding time requirements.

Burden means the total time, effort, or financial resources expended by persons to generate, maintain, retain, or disclose or provide information to or for a Federal agency. This includes the time needed to review instructions; develop, acquire, install, and utilize technology and systems for the purpose of collecting, validating, and verifying information, processing and maintaining information, and disclosing and providing information; adjust the existing ways to comply with any previously applicable instructions and requirements; train personnel to be able to respond to a collection of information; search data sources; complete and review the collection of information; and transmit or otherwise disclose the information.

An Agency may not conduct or sponsor, and a person is not required to respond to a collection of information unless it displays a currently valid OMB control number. The OMB control numbers for EPA's regulations in 40 CFR are listed in 40 CFR Part 9.

C. Regulatory Flexibility Act

The RFA generally requires an agency to prepare a regulatory flexibility analysis of any rule subject to notice and comment rulemaking requirements under the Administrative Procedure Act or any other statute unless the agency certifies that the rule will not have a significant economic impact on a substantial number of small entities. Small entities include small businesses, small organizations, and small governmental jurisdictions.

For purposes of assessing the impacts of this rule on small entities for methods under the Clean Water Act, small entity is defined as: (1) A small business as defined by the Small Business Administration's (SBA) regulations at 13 CFR 121.201; (2) a small governmental jurisdiction that is a government of a city, county, town, school district or special district with a population less than 50,000; and (3) a small organization that is any not-for-profit enterprise which is independently owned and operated and is not dominant in its field. The RFA provides default definitions for each type of small entity. It also authorizes an agency to use alternative definitions for each category of small entity, ''which are appropriate to the activities of the agency'' after proposing the alternative definition(s) in the **Federal Register** and taking comment (5 U.S.C. 601(3)–(5)). In addition to the above, to establish an alternative small business definition, agencies must consult with SBA's Chief Counsel for Advocacy.

For purposes of assessing the impacts of this rule on small entities for methods under the Safe Drinking Water Act, EPA considered small entities to be public water systems serving 10,000 or fewer persons. This is the cut-off level specified by Congress in the 1996 Amendments to the Safe Drinking Water Act for small system flexibility provisions. In accordance with the RFA requirements, EPA proposed using this alternative definition in the **Federal Register** (63 FR 7620, February 13, 1998), requested public comment, consulted with the Small Business Administration, and expressed its intention to use the alternative definition for all future drinking water regulations in the Consumer Confidence Reports regulation (63 FR 44511, August 19, 1998). As stated in that final rule, the alternative definition would be applied to this regulation as well.

After considering the economic impacts of today's final rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. This action approves new and updated versions of testing procedures, withdraws some older testing procedures, and approves new sample collection, preservation, and holding time requirements. Generally, these changes will have a positive impact on small entities by increasing method flexibility, thereby allowing entities to reduce costs by choosing more cost-effective methods. In some cases, analytical costs may increase slightly due to the additional QC requirements included in the methods that are being approved to replace older EPA methods. However, most laboratories that analyze samples for EPA compliance monitoring have already instituted QC requirements as part of their laboratory practices. We have determined that a small number of small entities that are still using the CFC–113 based oil and grease methods may need to devote resources to analyst training when they switch to hexane-based methods. However, due to the decreased availability of CFC–113 in the marketplace, we anticipate that the cost differential, if any, will soon favor the use of the hexane-based methods. The phaseout of CFC–113 based methods is required to comply with the Montreal Protocol which prohibits the use of CFC–113 based methods after December 31, 2005.

Although this final rule will not have a significant economic impact on a substantial number of small entities, EPA nonetheless has tried to reduce the impact of this rule on small entities. Anticipating the prohibition of CFC–113 based methods, EPA promulgated hexane-based methods in May 1999. EPA has determined that most laboratories have now switched to hexane-based oil and grease methods, making the analysis costs competitive with the CFC–113 based methods.

D. Unfunded Mandates Reform Act

Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), Public Law 104–4, establishes requirements for Federal agencies to assess the effects of their regulatory actions on State, Tribal, and local governments and the private sector. Under section 202 of the UMRA, EPA generally must prepare a written statement, including a cost-benefit analysis, for proposed and final rules with ''Federal mandates'' that may result in expenditures to State, local, and Tribal governments, in the aggregate, or to the private sector, of $100 million or more in any one year. Before promulgating an EPA rule for which a written statement is needed, section 205 of the UMRA generally requires EPA to identify and consider a reasonable number of regulatory alternatives and adopt the least costly, most cost-effective or least burdensome alternative that achieves the objectives of the rule. The provisions of section 205 do not apply when they are inconsistent with applicable law. Moreover, section 205 allows EPA to adopt an alternative other than the least costly, most cost-effective or least burdensome alternative if the Administrator publishes with the final rule an explanation of why that alternative was not adopted.

Before EPA establishes any regulatory requirements that may significantly or uniquely affect small governments, including Tribal governments, it must have developed under section 203 of the UMRA a small government agency plan. The plan must provide for the notification of potentially affected small governments, enabling officials of affected small governments to have meaningful and timely input in the development of EPA regulatory proposals with significant Federal intergovernmental mandates, and informing, educating, and advising small governments on compliance with the regulatory requirements.

This rule contains no Federal mandates (under the regulatory provisions of Title II of UMRA) for State, local, or Tribal governments or the private sector. The rule imposes no enforceable duty on any State, local, or Tribal governments or the private sector. In fact, this rule should (on the whole) save money for governments and the private sector by increasing method flexibility, and allowing these entities to reduce monitoring costs by taking advantage of innovations. Thus, today's rule is not subject to the requirements of Sections 202 and 205 of the UMRA.

EPA has determined that this rule contains no regulatory requirements that might significantly or uniquely affect small governments. Generally, this action will have a positive impact by increasing method flexibility, thereby allowing method users to reduce costs by choosing more cost effective methods. In some cases, analytical costs may increase slightly due to changes in methods, but these increases are neither significant nor unique to small governments. This rule merely approves new and updated versions of testing procedures, withdraws some older testing procedures, and approves new sample collection, preservation, and holding time requirements. Thus, today's rule is not subject to the requirements of Section 203 of UMRA.

E. Executive Order 13132: Federalism

Executive Order 13132, entitled ''Federalism'' (64 FR 43255, August 10, 1999), requires EPA to develop an accountable process to ensure ''meaningful and timely input by State and local officials in the development of regulatory policies that have federalism implications.'' ''Policies that have federalism implications'' is defined in the Executive Order to include regulations that have ''substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government.''

This final rule does not have federalism implications. It will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132. This rule merely approves new and updated versions of testing procedures, withdraws some older testing procedures, and approves new sample collection, preservation, and holding time requirements. The costs to State and local governments will be minimal (in fact, governments may see a cost savings), and the rule does not preempt State law. Thus, Executive Order 13132 does not apply to this rule.

In the spirit of Executive Order 13132, and consistent with EPA policy to promote communications between EPA and State and local governments, EPA specifically solicited comment on the proposed rule from State and local officials.

F. Executive Order 13175: Consultation and Coordination With Indian Tribal Governments

Executive Order 13175, entitled ''Consultation and Coordination with Indian Tribal Governments'' (65 FR 67249, November 9, 2000), requires EPA to develop an accountable process to ensure ''meaningful and timely input by tribal officials in the development of regulatory policies that have tribal implications.''

This final rule does not have tribal implications, as specified in Executive Order 13175. It will not have substantial direct effects on Tribal governments, on the relationship between the Federal government and Indian tribes, or on the distribution of power and responsibilities between the Federal government and Indian tribes. This rule merely approves new and updated versions of testing procedures, withdraws some older testing procedures, and approves new sample collection, preservation, and holding time requirements. The costs to Tribal governments will be minimal (in fact, governments may see a cost savings), and the rule does not preempt State law. Thus, Executive Order 13175 does not apply to this rule.

G. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks

Executive Order 13045: ''Protection of Children from Environmental Health Risks and Safety Risks'' (62 FR 19885, April 23, 1997) applies to any rule that: (1) Is determined to be ''economically significant'' as defined under Executive Order 12866, and (2) concerns an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children. If the regulatory action meets both criteria, the Agency must evaluate the environmental health or safety effects of the planned rule on children, and explain why the planned regulation is preferable to other potentially effective and reasonably feasible alternatives considered by the Agency. This final rule is not subject to the Executive Order 13045 because it is not economically significant as defined in Executive Order 12866. Further it does not concern an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children. This action approves new and updated versions of testing procedures, withdraws some older testing procedures, and approves new sample collection, preservation, and holding time requirements.

H. Executive Order 13211: Actions That Significantly Affect Energy Supply, Distribution, or Use

This rule is not subject to Executive Order 13211, ''Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use'' (66 FR 28355 (May 22, 2001)) because it is not a significant regulatory action under Executive Order 12866.

I. National Technology Transfer and Advancement Act

Section 12(d) of the National Technology Transfer and Advancement Act of 1995, (NTTAA), Public Law 104–113, section 12(d) (15 U.S.C. 272 note), directs EPA to use voluntary consensus standards in its regulatory activities unless to do so would be inconsistent with applicable law or otherwise impractical. Voluntary consensus standards are technical standards (e.g., material specifications, test methods, sampling procedures, and business practices) that are developed or adopted by voluntary consensus standard bodies. The NTTAA directs EPA to provide Congress, through the OMB, explanations when the Agency decides not to use available and applicable voluntary consensus standards. This final rule approves the use of over 150 standards developed by Standard Methods and ASTM International for use in compliance monitoring.

J. Congressional Review Act

The Congressional Review Act, 5 U.S.C. Section 801 *et seq.*, as added by the Small Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States prior to publication of the rule in the **Federal Register**. A major rule cannot take effect until 60 days after it is published in the **Federal Register**. This action is not a ''major rule'' as defined by 5 U.S. C Section 804(2). This rule will be effective April 11, 2007.

List of Subjects

40 CFR Part 122

Administrative practice and procedure, Confidential business information, Environmental protection, Hazardous substances, Reporting and recordkeeping requirements, Water pollution control.

40 CFR Part 136

Environmental protection, Incorporation by reference, Reporting and recordkeeping requirements, Water pollution control.

40 CFR Part 141

Chemicals, Environmental protection, Incorporation by reference, Indians-lands, Intergovernmental relations, Radiation Protection, Reporting and recordkeeping requirements, Water supply.

40 CFR Part 143

Chemicals, Environmental protection, Incorporation by reference, Indians-lands, Water supply.

40 CFR Part 430

Environmental protection, Paper and paper products industry, Reporting and recordkeeping requirements, Waste treatment and disposal, Water pollution control.

40 CFR Part 455

Chemicals, Environmental protection, Packaging and containers, Pesticides and pests, Waste treatment and disposal, Water pollution control.

40 CFR Part 465

Coil coating industry, Environmental protection, Reporting and recordkeeping requirements, Waste treatment and disposal, Water pollution control.

Dated: July 21, 2006.

Stephen L. Johnson,

Administrator.

■ For the reasons set out in the preamble, title 40, chapter I of the Code of Federal Regulations, is amended as follows:

PART 122—EPA ADMINISTERED PERMIT PROGRAMS: THE NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

■ 1. The authority citation for Part 122 continues to read as follows:

Authority: The Clean Water Act, 33 U.S.C. 1251 *et seq.*

■ 2. Section 122.1 is amended by revising paragraph (a)(4) to read as follows:

§ 122.1 Purpose and Scope.

(a) * * *

(4) Regulatory provisions in Parts 125, 129, 133, 136 of this chapter and 40 CFR subchapter N and subchapter O of this chapter also implement the NPDES permit program.

* * * * *

■ 3. Section 122.21 is amended as follows:

■ a. By revising paragraph (g)(7)(i).

■ b. By revising introductory text in paragraph (h)(4)(i).

■ c. By revising paragraph (j)(4)(viii).

§ 122.21 Application for a permit (applicable to State programs, see § 123.25)

* * * * *

(g) * * *

(7) *Effluent characteristics.* (i) Information on the discharge of pollutants specified in this paragraph (g)(7) (except information on storm water discharges which is to be provided as specified in § 122.26). When ''quantitative data'' for a pollutant are required, the applicant must collect a sample of effluent and analyze it for the pollutant in accordance with analytical methods approved under Part 136 of this chapter unless use of another method is required for the pollutant under 40 CFR subchapters N or O. When no analytical method is approved under Part 136 or required under subchapters N or O, the applicant may use any suitable method but must provide a description of the method. When an applicant has two or more outfalls with substantially identical effluents, the Director may allow the applicant to test only one outfall and report that quantitative data as applying to the substantially identical outfall. The requirements in paragraphs (g)(7)(vi) and (vii) of this section state that an applicant must provide quantitative data for certain pollutants known or believed to be present do not apply to pollutants present in a discharge solely as the result of their presence in intake water; however, an applicant must report such pollutants as present. When paragraph (g)(7) of this section requires analysis of pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, fecal coliform (including *E. coli*), and Enterococci (previously known as fecal streptococcus at § 122.26 (d)(2)(iii)(A)(3)), or volatile organics, grab samples must be collected for those pollutants. For all other pollutants, a 24-hour composite sample, using a minimum of four (4) grab samples, must be used unless specified otherwise at 40 CFR Part 136. However, a minimum of one grab sample may be taken for effluents from holding ponds or other impoundments with a retention period greater than 24 hours. In addition, for discharges other than storm water discharges, the Director may waive composite sampling for any outfall for which the applicant demonstrates that the use of an automatic sampler is infeasible and that the minimum of four (4) grab samples will be a representative sample of the effluent being discharged. Results of analyses of individual grab samples for any parameter may be averaged to obtain the daily average. Grab samples that are not required to be analyzed immediately (see Table II at 40 CFR 136.3 (e)) may be composited in the laboratory, provided that container, preservation, and holding time requirements are met (see Table II at 40 CFR 136.3 (e)) and that sample integrity is not compromised by compositing.

* * * * *

(h) * * *

(4) * * *

(i) Quantitative data for the pollutants or parameters listed below, unless testing is waived by the Director. The quantitative data may be data collected over the past 365 days, if they remain representative of current operations, and must include maximum daily value, average daily value, and number of measurements taken. The applicant must collect and analyze samples in accordance with 40 CFR Part 136. When analysis of pH, temperature, residual chlorine, oil and grease, or fecal coliform (including *E. coli*), and Enterococci (previously known as fecal streptococcus) and volatile organics is required in paragraphs (h)(4)(i)(A) through (K) of this section, grab samples must be collected for those pollutants. For all other pollutants, a 24-hour composite sample, using a minimum of four (4) grab samples, must be used unless specified otherwise at 40 CFR Part 136. For a composite sample, only one analysis of the composite of aliquots is required. New dischargers must include estimates for the pollutants or parameters listed below instead of actual sampling data, along with the source of each estimate. All levels must be reported or estimated as concentration and as total mass, except for flow, pH, and temperature.

* * * * *

(j) * * *

(4) * * *

(viii) Applicants must collect samples of effluent and analyze such samples for pollutants in accordance with analytical methods approved under 40 CFR Part 136 unless an alternative is specified in the existing NPDES permit. When analysis of pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, fecal coliform (including *E. coli*), or volatile organics is required in paragraphs (j)(4)(ii) through (iv) of this section, grab samples must be collected for those pollutants. For all other pollutants, 24-hour composite samples must be used. For a composite sample, only one analysis of the composite of aliquots is required.

* * * * *

■ 4. Section 122.41 is amended by revising paragraphs (j)(4) and (l)(4)(ii) to read as follows:

§ 122.41 Conditions applicable to all permits (applicable to State programs, see § 123.25).

* * * * *

(j) * * *

(4) Monitoring must be conducted according to test procedures approved under 40 CFR Part 136 unless another method is required under 40 CFR subchapters N or O.

* * * * *

(l) * * *

(4) * * *

(ii) If the permittee monitors any pollutant more frequently than required by the permit using test procedures approved under 40 CFR Part 136, or another method required for an industry-specific waste stream under 40 CFR subchapters N or O, the results of such monitoring shall be included in the calculation and reporting of the data submitted in the DMR or sludge reporting form specified by the Director.

* * * * *

■ 5. Section 122.44 is amended by revising paragraph (i)(1)(iv) to read as follows:

§ 122.44 Establishing limitations, standards, and other permit conditions (applicable to State NPDES programs; see § 123.25).

* * * * *

(i) * * *

(1) * * *

(iv) According to test procedures approved under 40 CFR Part 136 for the analyses of pollutants or another method is required under 40 CFR subchapters N or O. In the case of pollutants for which there are no approved methods under 40 CFR Part 136 or otherwise required under 40 CFR subchapters N or O, monitoring must be conducted according to a test procedure specified in the permit for such pollutants.

* * * * *

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

■ 6. The authority citation for Part 136 continues to read as follows:

Authority: Secs. 301, 304(h), 307, and 501(a) Pub. L. 95–217, 91 Stat. 1566, *et seq.* (33 U.S.C. 1251, *et seq.*) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977.)

■ 7. Section 136.3 is amended as follows:

■ a. In paragraph (a) by revising the introductory text and Tables IA, IB, IC, ID, and IE.

■ b. In paragraph (a) by adding Table IG after the notes of Table IF.

■ c. In paragraph (b) by revising references 6, 10, and 17, and adding references 63 through 69.

■ d. By revising paragraphs (c), (d), and (e).

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, and IG. In the event of a conflict between the reporting requirements of 40 CFR Parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of 40 CFR Parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, and IG. The incorporation by reference of these documents, as specified in paragraph (b) of this section, was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies of the documents may be obtained from the sources listed in paragraph (b) of this section. You can get information about obtaining these documents from the EPA Office of Water Engineering and Analysis Division at 202–566–1000. Documents may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue, NW., Washington, DC (Telephone: 202–566–2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html*

These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the **Federal Register**. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, IE, IF, and IG or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under certain circumstances (paragraph (b) or (c) of this section or 40 CFR 401.13) other test procedures may be used if such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and the Director of the State in which such discharge will occur does not object to the use of an additional or alternate test procedure.

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS

Parameter and units	Method [1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods online	AOAC, ASTM, USGS	Other
Bacteria:						
1. Coliform (fecal), number per 100 mL.	Most Probable Number (MPN), 5 tube 3 dilution, or	p. 132 [3]	9221C or E	9221C or E–99.		
	Membrane filter (MF) [2], single step.	p. 124 [3]	9222D	9222D–97	B–0050–85 [5].	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132 [3]	9221C or E	9221C or E–99.		
	MF, single step [6]	p. 124 [3]	9222D	9222D–97.		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B	9221B–99.		
	MF [2], single step or two step.	p. 108 [3]	9222B	9222B–97	B–0025–85 [5].	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B	9221B–99.		
	MF [2] with enrichment	p. 111 [3]	9222 (B+B.5c)	9222 (B+B.5c)–97.		
5. E. coli, number per 100 mL [28].	MPN [7, 9, 15], multiple tube,		9221B.1/ 9221F [12 14].	9221B.1–99/ 9221F [12 14].		

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS—Continued

Parameter and units	Method[1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods online	AOAC, ASTM, USGS	Other
	multiple tube/multiple well,	…	9223B[13]	9223B–97[13]	991.15[11]	Colilert®[13,17], Colilert-18®[13,16,17]
	MF two step, or	1103.1[20]	9222B/9222G[19], 9213D.	9222B–97/ 9222G[19].	D5392–93[10].	
	single step	1603[21], 1604[22]	…	…	…	mColiBlue-24®[18]
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution,	p. 139[3]	9230B	9230B–93.		
	MF[2], or	p. 136[3]	9230C	9230C–93	B–0055–85[5].	
	Plate count	p. 143[3].				
7. Enterococci, number per 100 mL[28].	MPN[7,9] multiple tube,	…	9230B	9230B–93.		
	multiple tube/multiple well.	…	…	…	D6503–99[10].	Entero-lert®[13,23]
	MF[2,6,7,8,9], two step	1106.1[24]	9230C	9230C–93	D5259–92[10].	
	single step, or	1600[25].				
	Plate count	p. 143[3].				
Protozoa:						
8. *Cryptosporidium*[28].	Filtration/IMS/FA	1622[26], 1623[27].				
9. *Giardia*[28]	Filtration/IMS/FA	1623[27].				
Aquatic Toxicity:						
10. Toxicity, acute, fresh water organisms, LC_{50}, percent effluent.	*Ceriodaphnia dubia* acute.	2002.0[29].				
	Daphnia puplex and *Daphnia magna* acute.	2021.0[29].				
	Fathead Minnow, *Pimephales promelas,* and Bannerfin shiner, *Cyprinella leedsi,* acute.	2000.0[29].				
	Rainbow Trout, *Oncorhynchus mykiss,* and brook trout, *Salvelinus fontinalis,* acute.	2019.0[29].				
11. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC_{50}, percent effluent.	Mysid, *Mysidopsis bahia,* acute.	2007.0[29].				
	Sheepshead Minnow, *Cyprinodon variegatus,* acute.	2004.0[29].				
	Silverside, *Menidia beryllina, Menidia menidia,* and *Menidia peninsulae,* acute.	2006.0[29].				
12. Toxicity, chronic, fresh water organisms, NOEC or IC_{25}, percent effluent.	Fathead minnow, *Pimephales promelas,* larval survival and growth.	1000.0[30].				
	Fathead minnow, *Pimephales promelas,* embryo-larval survival and teratogenicity.	1001.0[30].				
	Daphnia, *Ceriodaphnia dubia,* survival and reproduction.	1002.0[30].				

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS—Continued

Parameter and units	Method[1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods online	AOAC, ASTM, USGS	Other
	Green alga, *Selenastrum capricornutum,* growth.	1003.0[30].				
13. 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[31].				
	Sheepshead minnow, *Cyprinodon variegatus,* embryo-larval survival and teratogenicity.	1005.0[31].				
	Inland silverside, *Menidia beryllina,* larval survival and growth.	1006.0[31].				
	Mysid, *Mysidopsis bahia,* survival, growth, and fecundity.	1007.0[31].				
	Sea urchin, *Arbacia punctulata,* fertilization.	1008.0[31].				

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

[2] A 0.45-μ 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] USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH, EPA/600/8–78/017.

[4] [Reserved]

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

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

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

[8] When the MF method has not been used previously to test ambient waters with high turbidity, large number of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

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

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

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

[12] The multiple-tube fermentation test is used in 9221B.1. 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, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 μg/mL of MUG may be used.

[15] 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.

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

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

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

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

[20] USEPA. 2002. Method 1103.1: *Escherichia coli* (*E. coli*) In Water By Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–02–020.

[21] USEPA. 2002. Method 1603: *Escherichia coli* (*E. coli*) In Water By Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–02–023.

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

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

[24] USEPA. 2002. Method 1106.1: Enterococci In Water By Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE–EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–02–021.

[25] USEPA. 2002. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–02–022.

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

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

[28] Recommended for enumeration of target organism in ambient water only.

[29] USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA/821/R–02/012.

[30] USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Fourth Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA/821/R–02/013.

[31] USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA/821/R–02/014.

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology[58]	Reference (method number or page)					
		EPA[35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
1. Acidity, as CaCO$_3$, mg/L	Electrometric endpoint or phenolphthalein endpoint.		2310 B(4a)	2310 B(4a)	2310 B(4a)–97	D1067–92, 02	I–1020–85[2]
2. Alkalinity, as CaCO$_3$, mg/L	Electrometric or Colorimetric titration to pH 4.5, manual, or		2320 B	2320 B	2320 B–97	D1067–92, 02	973.43[3], I–1030–85[2]
	automatic	310.2 (Rev. 1974)[1].					I–2030–85[2]
3. Aluminum—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 D		3111 D–99		I–3051–85[2]
	AA furnace		3113 B		3113 B–99.		
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–9750
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	Direct Current Plasma (DCP)[36]					D4190–94, 99	See footnote[34]
	Colorimetric (Eriochrome cyanine R)		3500–Al D	3500–Al B	3500–Al B–01.		
4. Ammonia (as N), mg/L	Manual, distillation (at pH 9.5)[6] followed by:	350.1, Rev. 2.0 (1993).	4500–NH B$_3$	4500–NH$_3$ B	4500–NH$_3$ B–97		973.49[3]
	Nesslerization		4500–NH$_3$ C (18th only).			D1426–98, 03 (A)	973.49[3], I–3520–85[2]
	Titration		4500–NH$_3$ C (19th) and 4500–NH$_3$ E (18th).	4500–NH$_3$ C	4500–NH$_3$ C–97.		
	Electrode		4500–NH$_3$ D or E (19th) and 4500–NH$_3$ F or G (18th).	4500–NH$_3$ D or E	4500–NH$_3$ D or E–97.	D1426–98, 03 (B).	
	Automated phenate, or	350.1[60], Rev. 2.0 (1993).	4500–NH$_3$ G (19th) and 4500–NH$_3$ H (18th).	4500–NH$_3$ G	4500–NH$_3$ G–97		I–4523–85[2]
	Automated electrode						See footnote 7
	Ion Chromatography					D6919–03.	
5. Antimony—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B		3111 B–99.		
	AA furnace		3113 B		3113 B–99.		
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
6. Arsenic—Total,[4] mg/L	Digestion[4] followed by	206.5 (Issued 1978)[1].					
	AA gaseous hydride		3114 B 4.d		3114 B 4.d–97	D2972–97, 03 (B)	I–3062–85[2]
	AA furnace		3113 B		3113 B–99	D2972–97, 03 (C)	I–4063–98[49]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	Colorimetric (SDDC)		3500–As C	3500–As B	3500–As B–97	D2972–97, 03 (A)	I–3060–85
7. Barium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 D		3111 D–99		I–3084–85[2]
	AA furnace		3113 B		3113 B–99	D4382–95, 02.	

	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP[36]						See footnote[34]
8. Beryllium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99	D3645–93 (88), 03 (A).	I–3095–85[2]
	AA furnace		3113 B		3113 B–99	D3645–93 (88), 03 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP, or					D4190–94, 99	See footnote[34]
	Colorimetric (aluminon)		3500–Be D.				
9. Biochemical oxygen demand (BOD$_5$), mg/L.	Dissolved Oxygen Depletion		5210 B	5210 B	5210 B–01		973.44,[3] p. 17.[9], I–1578–78[8]
10. Boron—Total,[37] mg/L	Colorimetric (curcumin)		4500–B B	4500–B B	4500–B B–00		I–3112–85[2]
	ICP/AES, or	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B 99		I–4471–97[50]
	DCP					D4190–94, 99	See footnote 34
11. Bromide, mg/L	Titrimetric					D1246–95, 99 (C)	p. S44.[10]
							I–1125–85[2]
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						D6508, Rev. 2[54]
12. Cadmium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D3557–95, 02 (A or B).	974.27,[3] p. 37.[9], I–3135–85[2] or I–3136–85[2]
	AA furnace		3113 B		3113 B–99	D3557–95, 02 (D)	I–4138–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–1472–85[2] or I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP[36]					D4190–94, 99	See footnote[34]
	Voltametry[11], or					D3557–95, 02 (C).	
	Colorimetric (Dithizone)		3500–Cd D.				
13. Calcium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99	D511–93, 03(B)	I–3152–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	DCP, or						See footnote[34]
	Titrimetric (EDTA)		3500–Ca D	3500–Ca B	3500–Ca B–97	D511–93, 03(A).	
	Ion Chromatography					D6919–03.	
14. Carbonaceous biochemical oxygen demand (CBOD$_5$), mg/L[12].	Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B	5210 B	5210 B–01.		
15. Chemical oxygen demand (COD), mg/L	Titrimetric	410.3 (Rev. 1978)[1].	5220 C	5220 C	5220 C–97	D1252–95, 00 (A)	973.46[3], p. 17[9] I–3560–85[2]
	Spectrophotometric, manual or automatic.	410.4, Rev. 2.0 (1993).	5220 D	5220 D	5220 D–97	D1252–95, 00 (B)	See footnotes[13, 14]. I–3561–85[2]
16. Chloride, mg/L	Titrimetric: (silver nitrate) or		4500–Cl–B	4500–Cl–B	4500–Cl–B–97	D512–89(99) (B)	I–1183–85[2]
	(Mercuric nitrate)		4500–Cl–C	4500–Cl–C	4500–Cl–C–97	D512–89 (99) (A)	973.51[3], I–1184–85[2]
	Colorimetric: manual or						I–1187–85[2]
	Automated (Ferricyanide)		4500–Cl–E	4500–Cl–E	4500–Cl–E–97		I–2187–85[2]
	Potentiometric Titration		4500–Cl–D	4500–Cl–D	4500–Cl–D–97.		
	Ion Selective Electrode					D512–89(99)(C).	

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	Reference (method number or page)					
		EPA[35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						D6508, Rev. 2[54]
17. Chlorine—Total residual, mg/L; Titrimetric	Amperometric direct, or		4500–Cl D	4500–Cl D	4500–Cl D–00	D1253–86 (96), 03.	
	Amperometric direct (low level)		4500–Cl E	4500–Cl E	4500–Cl E–00.		
	Iodometric direct		4500–Cl B	4500–Cl B	4500–Cl B–00.		
	Back titration ether end–point[15] or		4500–Cl C	4500–Cl C	4500–Cl C–00.		
	DPD–FAS		4500–Cl F	4500–Cl F	4500–Cl F–00.		
	Spectrophotometric, DPD or		4500–Cl G	4500–Cl G	4500–Cl G–00.		
	Electrode						See footnote[16]
18. Chromium VI dissolved, mg/L	0.45–micron Filtration followed by:						
	AA chelation–extraction or		3111 C		3111 C–99		I–1232–85
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500–Cr E	3500–Cr C	3500–Cr C–01	D5257–97	993.23
	Colorimetric (Diphenyl–carbazide)		3500–Cr D	3500–Cr B	3500–Cr B–01	D1687–92, 02 (A)	I–1230–85
19. Chromium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B		3111 B–99	D1687–92, 02 (B)	974.27[3], I–3236–85[2]
	AA chelation–extraction		3111 C		3111 C–99.		
	AA furnace		3113 B		3113 B–99	D1687–92, 02 (C)	I–3233–93[46]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP,[36] or					D4190–94, 99	See footnote[34]
	Colorimetric (Diphenyl–carbazide)		3500–Cr D	3500–Cr B	3500–Cr B–01.		
20. Cobalt—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B or C		3111 B or C–99	D3558–94, 03 (A or B).	p. 37[9], I–3239–85[2]
	AA furnace		3113 B		3113 B–99	D3558–94, 03 (C)	I–4243–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP					D4190–94, 99	See footnote[34]
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity.	Colorimetric (ADMI), or		2120 E	2120 E			See footnote[18]
	(Platinum cobalt), or		2120 B	2120 B	2120 B–01		I–1250–85[2]
	Spectrophotometric		2120 C	2120 C.			
22. Copper—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D1688–95, 02 (A or B).	974.27[3] p. 37[9] I–3270–85[2] or I–3271–85[2]
	AA furnace		3113 B		3113 B–99	D1688–95, 02 (C)	I–4274–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP[36] or					D4190–94, 99	See footnote[34]
	Colorimetric (Neocuproine) or		3500–Cu D	3500–Cu B	3500–Cu B–99.		
	(Bicinchoninate)		3500–Cu E	3500–Cu C	3500–Cu C–99		See footnote[19]

23. Cyanide—Total, mg/L	Automated Distillation and Colorimetry, or.						Kelada–01 [55]
	Manual distillation with $MgCl_2$ followed by:	335.4, Rev. 1.0 (1993) [57].	4500–CN^-C	4500–CN^-C		D2036–98(A)	10–204–00–1–X [56]
	Titrimetric or		4500–CN^-D	4500–CN^-D	4500–CN^-D–99		p. 22 [9]
	Spectrophotometric, manual or		4500–CN^-E	4500–CN^-E	4500–CN^-E–99	D2036–98(A)	I–3300–85
	Automated [20] or	335.4, Rev. 1.0 (1993) [57].					10–204–00–1–X [56], I–4302–85 [2]
	Ion Selective Electrode		4500–CN^-F	4500–CN^-F	4500–CN^-F–99	D2036–98(A).	
24. Available Cyanide, mg/L	Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$ followed by Titrimetric or Spectrophotometric.		4500–CN^-G	4500–CN^-G	4500–CN^-G–99	D2036–98(B).	
	Flow injection and ligand exchange, followed by amperometry [61].					D6888–04	OIA–1677 [44]
	Automated Distillation and Colorimetry.						Kelada–01 [55]
25. Fluoride—Total, mg/L	Manual distillation [6] followed by:		4500–F^-B	4500–F^-B	4500–F^-B–97.		
	Electrode, manual or		4500–F^-B	4500–F^-B	4500–F^-C–97	D1179–93, 99 (B).	
	Automated						I–4327–85 [2]
	Colorimetric, (SPADNS) or		4500–F^-D	4500–F^-D	4500–F^-D–97	D1179–93, 99 (A).	
	Automated complexone		4500–F^-E	4500–F^-E	4500–F^-E–97.		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97,03	993.30 [3]
	CIE/UV						D6508, Rev. 2 [54]
26. Gold—Total, [4] mg/L	Digestion [4] followed by:						
	AA direct aspiration, or		3111 B		3111 B–99.		
	AA furnace, or	231.2 (Rev. 1978) [1].					
	DCP						See footnote [34]
27. Hardness—Total, as $CaCO_3$, mg/L	Automated colorimetric,	130.1 (Issued 1971) [1].					
	Titrimetric (EDTA) or		2340 B or C	2340 B or C	2340 B or C–97	D1126–86(92), 02	973.5 2B [3], I–1338–85 [2]
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33)..						
28. Hydrogen ion (pH), pH units	Electrometric measurement or		4500–H^+ B	4500–H^+ B	4500–H^+ B–00	D1293–84 (90), 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:						
	AA direct aspiration or		3111 B		3111 B–99.		
	AA furnace	235.2 (Issued 1978) [1].					
30. Iron—Total, [4] mg/L	Digestion [4] followed by:						
	AA direct aspiration [36]		3111 B or C		3111 B or C–99	D1068–96, 03 (A or B).	974.27 [3], I–3381–85 [2]
	AA furnace		3113 B		3113 B–99	D1068–96, 03 (C).	
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97 [50]
	DCP [36] or					D4190–94, 99	See footnote [34]
	Colorimetric (Phenanthroline)		3500–Fe D	3500–Fe B	3500–Fe B–97	D1068–96, 03 (D)	See footnote [22]
31. Kjeldahl Nitrogen [5]—Total, (as N), mg/L	Digestion and distillation followed by: [20]		4500–N_{org} B or C and 4500–NH_3 B.	4500–N_{org} B or C and 4500–NH_3 B.	4500–N_{org} B or C–97 and 4500–NH_3 B–97.	D3590–89, 02 (A).	
	Titration or		4500–NH_3 C (19th) and 4500–NH_3 E (18th).	4500–NH_3 C	4500–NH_3 C–97	D3590–89, 02 (A)	973.48 [3]

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	Reference (method number or page)					
		EPA[35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
	Nesslerization or		4500–NH_3 C (18th Only).			D3590–89, 02 (A).	
	Electrode		4500–NH_3 F or G (18th) and 4500–NH_3 D or E (19th).	4500–NH_3 D or E	4500–NH_3 D or E–97.		
	Automated phenate colorimetric	351.1 (Rev. 1978)[1].					I–4551–78[8]
	Semi-automated block digestor colorimetric.	351.2, Rev. 2.0 (1993).				D3590–89, 02 (B)	I–4515–91[45]
	Manual or block digestor potentiometric.					D3590–89, 02 (A).	
	Block digester, followed by Auto distillation and Titration, or.						See footnote[39]
	Nesslerization, or						See footnote[40]
	Flow injection gas diffusion						See footnote[41]
32. Lead—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D3559–96, 03 (A or B).	974.27[3], I–3399–85[2]
	AA furnace		3113 B		3113 B–99	D3559–96, 03 (D)	I–4403–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP[36]					D4190–94, 99	See footnote[34]
	Voltametry[11] or					D3559–96, 03 (C).	
	Colorimetric (Dithizone)		3500–Pb D	3500–Pb B	3500–Pb B–97.		
33. Magnesium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99	D511–93, 03(B)	974.27[3], I–3447–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	DCP or						See footnote[34]
	Gravimetric		3500–Mg D.				
	Ion Chromatography					D6919–03.	
34. Manganese—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B		3111 B–99	D858–95, 02 (A or B).	974.27[3], I–3454–85[2]
	AA furnace		3113 B		3113 B–99	D858–95, 02 (C).	
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP36, or					D4190–94, 99	See footnote[34]
	Colorimetric (Persulfate), or		3500—Mn D	3500–Mn B	3500–Mn B–99		920.203[3]
	(Periodate)						See footnote[23]
35. Mercury—Total[4], mg/L	Cold vapor, manual or	245.1, Rev. 3.0 (1994).	3112 B		3112 B–99	D3223–97, 02	977.22[3], I–3462–85[2]
	Automated	245.2 (Issued 1974).					
	Cold vapor atomic fluorescence spectrometry (CVAFS).	245.7 Rev. 2.0 (2005)[59].					
	Purge and Trap CVAFS	1631E[43].					
36. Molybdenum—Total[4], mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99		I–3490–85[2]

	AA furnace		3113 B		3113 B–99		I–3492–96[47]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP						See footnote[34]
37. Nickel—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D1886–90, 94 (98) (A or B).	I–3499–85[2]
	AA furnace		3113 B		3113 B–99	D1886–90, 94 (98) (C).	I–4503–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP[36], or					D4190–94, 99	See footnote[34]
	Colorimetric (heptoxime)		3500–Ni D (17th Edition).				
38. Nitrate (as N), mg/L	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						D6508, Rev. 2[54]
	Ion Selective Electrode		4500–NO_3^-D	4500–NO_3^-D	4500–NO_3^-D–00.		
	Colorimetric (Brucine sulfate), or	352.1[1]					973.50[3], 419D[1, 7], p. 28[9]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40)..						
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction, manual or		4500–NO_3^-E	4500–NO_3^-E	4500–NO3–E–00	D3867–99(B).	
	Automated, or	353.2, Rev. 2.0 (1993).	4500–NO_3^-F	4500–NO_3^-F	4500–NO_3^-F–00	D3867–99(A)	I–4545–85[2]
	Automated hydrazine		4500–NO_3^-H	4500–NO_3^-H	4500–NO_3^-H–00.		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97	993.30[3]
	CIE/UV						D6508, Rev. 2[54]
40. Nitrite (as N), mg/L	Spectrophotometric: Manual or		4500–NO_2^-B	4500–NO_2^-B	4500–NO_2^-B–00		See footnote[25]
	Automated (Diazotization)						I–4540–85[2]
	Automated (*bypass cadmium reduction).	353.2, Rev. 2.0 (1993).	4500–NO_3^-F	4500–NO_3^-F	4500–NO_3^-F–00	D3867–99(A)	I–4545–85[2]
	Manual (*bypass cadmium reduction)		4500–NO_3^-E	4500–NO_3^-E	4500–NO_3^-E–00	D3867–99(B).	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						D6508, Rev.2[54]
41. Oil and grease—Total recoverable, mg/L	Hexane extractable material (HEM): n–Hexane extraction and gravimetry.	1664A[42]		5520 B[38]	5520 B–01[38].		
	Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry..	1664A[42].					
42. Organic carbon—Total (TOC), mg/L	Combustion or oxidation		5310 B, C, or D	5310 B, C, or D	5310 B, C, or D–00	D2579–93 (A or B)	973.47,[3] p. 14[24]
43. Organic nitrogen (as N), mg/L	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4).						
44. Orthophosphate (as P), mg/L	Ascorbic acid method:.						
	Automated, or	365.1, Rev. 2.0 (1993).	4500–P F	4500–P F			973.56[3], I–4601–85[2]
	Manual single reagent		4500–P E	4500–P E		D515–88(A)	973.55[3]
	Manual two reagent	365.3 (Issued 1978)[1].					

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	Reference (method number or page)					
		EPA[35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						D6508, Rev. 2[54]
45. Osmium—Total[4], mg/L	Digestion[4] followed by:						
	AA direct aspiration, or		3111 D		3111 D–99.		
	AA furnace	252.2 (Issued 1978)[1].					
46. Oxygen, dissolved, mg/L	Winkler (Azide modification), or		4500–O C	4500–O C	4500–O C–01	D888–92, 03 (A)	973.4 5B[3], I–1575–78[8]
	Electrode		4500–O G	4500–O G	4500–O G–01	D888–92, 03 (B)	I–1576–78[8]
47. Palladium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration, or		3111 B		3111 B–99		p. S27[10]
	AA furnace	253.2[1] (Issued 1978).					p. S28[10]
	DCP						See footnote[34]
48. Phenols, mg/L	Manual distillation[26] Followed by:	420.1[1] (Rev. 1978).					See footnote[27]
	Colorimetric (4AAP) manual, or	420.1[1] (Rev. 1978).					See footnote[27]
	Automated	420.4 Rev. 1.0 (1993).					
49. Phosphorus (elemental), mg/L	Gas–liquid chromatography						See footnote[28]
50. Phosphorus—Total, mg/L	Persulfate digestion followed by:[20]		4500–P B.5	4500–P B.5			973.55[3]
	Manual or	365.3[1] (Issued 1978).	4500–P E	4500–P E		D515–88(A).	
	Automated ascorbic acid reduction	365.1 Rev. 2.0 (1993).	4500–P F	4500–P F			973.56[3], I–4600–85[2]
	Semi–automated block digestor	365.4[1] (Issued 1974).				D515–88(B)	I–4610–91[48]
51. Platinum—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99.		
	AA furnace	255.2[1].					
	DCP						See footnote[34]
52. Potassium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99		973.53[3], I–3630–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	Flame photometric, or		3500–K D	3500–K B	3500–K B–97.		
	Colorimetric						317 B[17]
	Ion Chromatography					D6919–03.	
53. Residue—Total, mg/L	Gravimetric, 103–105°		2540 B	2540 B	2540 B–97		I–3750–85[2]
54. Residue—filterable, mg/L	Gravimetric, 180°		2540 C	2540 C	2540 C–97		I–1750–85[2]
55. Residue—non–filterable (TSS), mg/L	Gravimetric, 103–105 °C post washing of residue.		2540 D	2540 D	2540 D–97		I–3765–85[2]
56. Residue—settleable, mg/L	Volumetric, (Imhoff cone), or gravimetric.		2540 F	2540 F	2540 F–97.		
57. Residue—Volatile, mg/L	Gravimetric, 550 °C	160.4[1]					I–3753–85[2]
58. Rhodium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration, or		3111 B		3111 B–99.		
	AA furnace	265.2[1].					
59. Ruthenium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration, or		3111 B		3111 B–99.		
	AA furnace	267.2[1].					
60. Selenium—Total,[4] mg/L	Digestion[4] followed by:						
	AA furnace		3113 B		3113 B–99	D3859–98, 03 (B)	I–4668–98[49]

	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	AA gaseous hydride		3114 B		3114 B–97	D3859–98, 03 (A)	I–3667–85[2]
61. Silica—Dissolved,[37] mg/L	0.45 micron filtration followed by:						
	Colorimetric, Manual or		4500–Si D	4500–SiO_2 C	4500–SiO_2C–97	D859–94, 00	I–1700–85[2]
	Automated (Molybdosilicate), or						I–2700–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
62. Silver—Total,[4, 31] mg/L	Digestion[4, 29] followed by:						
	AA direct aspiration		3111 B or C		3111 B or C–99		974.27[3], p. 37[9], I–3720–85[2]
	AA furnace		3113 B		3113 B–99		I–4724–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP						See footnote[34]
63. Sodium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99		973.54[3], I–3735–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	DCP, or						See footnote[34]
	Flame photometric		3500–Na D	3500–Na B	3500–Na B–97.		
	Ion Chromatography					D 6919–03.	
64. Specific conductance, micromhos/cm at 25°C.	Wheatstone bridge	120.1[1] (Rev. 1982).	2510 B	2510 B	2510 B–97	D1125–95 (99) (A)	973.40[3], I–2781–85[2]
65. Sulfate (as SO_4), mg/L	Automated colorimetric	375.2, Rev. 2.0 (1993).					
	Gravimetric		4500–SO_4^{2-}C or D	4500–SO_4^{2-}C or D			925.54[3]
	Turbidimetric					D516–90, 02	426C[30]
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						D6508, Rev. 2[54]
66. Sulfide (as S), mg/L	Titrimetric (iodine), or		4500–S^{2-}F (19th) 4500–S^{2-}E (18th).	4500–S^{2-}F	4500–S^{2-}F–00		I–3840–85[2]
	Colorimetric (methylene blue)		4500–S^{2-}D	4500–S^{2-}D	4500–S^{2-}D–00.		
	Ion Selective Electrode		4500–S^{2-}G	4500–S^{2-}G	4500–S^{2-}G–00	D4658–03.	
67. Sulfite (as SO_3), mg/L	Titrimetric (iodine-iodate)		4500–SO_3^{2-}B	4500–SO_3^{2-}B	4500–SO_3^{2-}B–00.		
68. Surfactants, mg/L	Colorimetric (methylene blue)		5540 C	5540 C	5540 C–00	D2330–88, 02.	
69. Temperature, °C	Thermometric		2550 B	2550 B	2550 B–00		See footnote[32]
70. Thallium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99.		
	AA furnace	279.2[1] (Issued 1978).					
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
71. Tin—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99		I–3850–78[8]
	AA furnace, or		3113 B		3113 B–99.		
	STGFAA	200.9, Rev. 2.2 (1994).					

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	Reference (method number or page)					
		EPA[35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
	ICP/AES	200.7, Rev. 4.4 (1994).					
72. Titanium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99.		
	AA furnace	283.2[1] (Issued 1978).					
	DCP						See footnote[34]
73. Turbidity, NTU[53]	Nephelometric	180.1, Rev. 2.0 (1993).	2130 B	2130 B	2130 B–01	D1889–94, 00	I–3860–85[2]
74. Vanadium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99.		
	AA furnace					D3373–93, 03.	
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP, or					D4190–94, 99	See footnote[34]
	Colorimetric (Gallic Acid)		3500–V D	3500–V B	3500–V B–97.		
75. Zinc –Total[4], mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D1691–95, 02 (A or B).	974.27[3], p. 37[9], I–3900–85[2]
	AA furnace	289.2[1] (Issued 1978).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99[59]		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]
	DCP,[36] or					D4190–94, 99	See footnote[34]
	Colorimetric (Dithizone) or		3500–Zn E.				
	(Zincon)		3500–Zn F	3500–Zn B	3500–Zn B–97		See footnote[33]

Table 1B Notes:

[1] "Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–CI), EPA–600/4–79–020 (NTIS PB 84–128677), Revised March 1983 and 1979 where applicable.

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

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

[4] 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 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.

[7] Ammonia, Automated Electrode Method, Industrial Method Number 379–75 WE, dated February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[8] The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).

[9] American National Standard on Photographic Processing Effluents, April 2, 1975. Available from ANSI, 25 West 43rd st., New York, NY 10036.

[10] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[11] 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, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

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

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

[16] Orion Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 mL 0.00281 N potassium iodate/100 mL solution, respectively.

[17] The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater,* 14th Edition, 1976.

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

[19] Copper, Biocinchoinate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

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

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

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

[23] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2–113 and 2–117, Hach Chemical Company, Loveland, CO 80537.

[24] Wershaw, R. L.,*et al.*, "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14.

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

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

[27] The approved method is cited in *Standard Methods for the Examination of Water and Wastewater,* 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576–81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrometric procedure.

[28] R.F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas–Liquid Chromatography," *Journal of Chromatography,* Vol. 47, No.3, pp. 421–426, 1970.

[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 approved method is that cited in *Standard Methods for the Examination of Water and Wastewater,* 15th Edition.

[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 I_2 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] Stevens, H.H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975.

[33] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2–231 and 2–333, Hach Chemical Company, Loveland, CO 80537.

[34] "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991, Thermo Jarrell Ash Corporation, 27 Forge Parkway, Franklin, MA 02038

[35] Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals."

[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", CEM Corporation, P.O. Box 200, Matthews, NC 28106–0200, April 16, 1992. Available from the CEM Corporation.

[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 extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Method 1664A). Use of other extraction solvents (*e.g.,* those in the 18th and 19th editions) is prohibited.

[39] Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[40] Nitrogen, Total Kjeldahl, Method PAI–DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[41] Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[42] Method 1664, Revision A "*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, February 1999. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.

[43] USEPA. 2001. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" September 2002, Office of Water, U.S. Environmental Protection Agency (EPA–821–R–02–024). The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA–821–R–96–011) are recommended to preclude contamination at low-level, trace metal determinations.

[44] Available Cyanide, Method OIA–1677, "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842–9010.

[45] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method," Open File Report (OFR) 00–170.

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

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

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

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

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

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

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

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

[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, 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" is available from Lachat Instruments 6645 W. Mill Road, Milwaukee, WI 53218, Telephone: 414–358–4200.

[57] When using sulfide removal test procedures described in Method 335.4, 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] Method 245.7, Rev. 2.0, "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry," February 2005, EPA–821–R–05–001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304, Telephone: 703–461–2100, Fax: 703–461–8056.

[60] The use of EDTA may decrease method sensitivity in some samples. Analysts may omit EDTA provided that all method specified quality control acceptance criteria are met.

[61] Samples analyzed for available cyanide using Methods OIA–1677 or D6888–04 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 analysis to no more than 30 minutes to preclude settling of materials in samples.

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter[1]	EPA method number[2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
1. Acenaphthene	610	625, 1625B	610	6440 B [18th, 19th, 20th].		D4657–92 (99)	See footnote[9], p. 27
2. Acenaphthylene	610	625, 1625B	610	6410 B, 6440 B, [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
3. Acrolein	603	624[4], 1624B.					
4. Acrylonitrile	603	624[4], 1624B.					
5. Anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
6. Benzene	602	624, 1624B		6200 B [20th] and 6210 B [18th,19th], 6200 C [20th] and 6220 B [18th,19th].	6200 B and C–97.		
7. Benzidine		625[5], 1625B	605				See footnote[3], p.1
8. Benzo(a)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
9. Benzo(a)pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
10. Benzo(b)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
11. Benzo(g,h,i) perylene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
12. Benzo(k) fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
13. Benzyl chloride							See footnote[3], p. 130: See footnote[6], p. S102
14. Benzyl butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
15. Bis(2-chloroethoxy) methane.	611	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
16. Bis(2-chloroethyl) ether	611	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
17. Bis(2-ethylhexyl) phthalate.	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
18. Bromodichloro-methane.	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97.		
19. Bromoform	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97.		
20. Bromomethane	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97.		
21. 4-Bromophenyl phenyl ether.	611	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
22. Carbon tetrachloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th].	6200 C–97		See footnote[3], p. 130
23. 4-Chloro-3-methyl phenol.	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote[9], p. 27
24. Chlorobenzene	601, 602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		See footnote[3], p. 130
25. Chloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
26. 2-Chloroethylvinyl ether.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter[1]	EPA method number[2, 7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
27. Chloroform	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		See footnote[3], p. 130
28. Chloromethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
29. 2-Chloronaph-thalene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
30. 2-Chlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B(00, 6420 B–00.		See footnote[9], p. 27
31. 4-Chlorophenyl phenyl ether.	611	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
32. Chrysene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
33. Dibenzo(a,h)anthracene.	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
34. Dibromochloro-methane.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
35. 1,2-Dichloro-benzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 C–97		See footnote[9], p. 27
36. 1,3-Dichloro-benzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 C–97		See footnote[9], p. 27
37. 1,4-Dichloro-benzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 C–97		See footnote[9], p. 27
38. 3,3-Dichloro-benzidine		625, 1625B	605	6410 B [18th, 19th, 20th].	6410 B–00.		
39. Dichlorodifluoro-methane.	601			6200 C [20th] and 6230 B [18th, 19th].	6200 C–97.		
40. 1,1-Dichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
41. 1,2-Dichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
42. 1,1-Dichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
43. trans-1,2-Dichloroethene.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
44. 2,4-Dichlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote[9], p. 27
45. 1,2-Dichloro-propane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
46. cis-1,3-Dichloro-propene.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number [2, 7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
47. trans-1,3-Dichloropropene.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
48. Diethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
49. 2,4-Dimethylphenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote [9], p. 27
50. Dimethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
51. Di-n-butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
52. Di-n-octyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
53. 2,3-Dinitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		
54. 2,4-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
55. 2,6-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
56. Epichlorohydrin							See footnote [3], p. 130; See footnote [6], p. S102
57. Ethylbenzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97		
58. Fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
59. Fluorene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
60. 1,2,3,4,6,7,8-Heptachlorodibenzofuran.		1613B [10].					
61. 1,2,3,4,7,8,9-Heptachlorodibenzofuran.		1613B [10].					
62. 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin.		1613B [10].					
63. Hexachlorobenzene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
64. Hexachloro-butadiene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
65. Hexachlorocyclopentadiene.	612	625 [5], 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
66. 1,2,3,4,7,8-Hexachlorodibenzofuran.		1613B [10].					
67. 1,2,3,6,7,8-Hexachlorodibenzofuran.		1613B [10].					
68. 1,2,3,7,8,9-Hexachlorodibenzofuran.		1613B [10].					
69. 2,3,4,6,7,8-Hexachlorodibenzofuran.		1613B [10].					
70. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin.		1613B [10].					
71. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin.		1613B [10].					
72. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin 1613B [10].		1613B [10].					
73. Hexachloroethane	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
74. Ideno(1,2,3-cd) pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
75. Isophorone	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
76. Methylene chloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th].	6200 C–97		See footnote [3], p. 130
77. 2-Methyl-4,6-dinitrophenol.	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote [9], p. 27
78. Naphthalene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter[1]	EPA method number[2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
79. Nitrobenzene	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
80. 2-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote[9], p. 27
81. 4-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote[9], p. 27
82. N-Nitrosodimethylamine.	607	6255, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
83. N-Nitrosodi-n-propylamine.	607	6255, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
84. N-Nitrosodiphenylamine.	607	6255, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[9], p. 27
85. Octachlorodibenzofuran.		1613B[10]*.					
86. Octachlorodibenzo-p-dioxin.		1613B[10].					
87. 2,2'-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether].	611	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00.		
88. PCB–1016	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 43; See footnote[8]
89. PCB–1221	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 43; See footnote[8]
90. PCB–1232	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 43; See footnote[8]
91. PCB–1242	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 43; See footnote[8]
92. PCB–1248	608	625.					
93. PCB–1254	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 43; See footnote[8]
94. PCB–1260	608	625		6410 B, 6630 B [18th, 19th, 20th].	6410 B–00		See footnote 3, p. 43; See footnote 8
95. 1,2,3,7,8-Pentachlorodibenzofuran .		1613B[10].					
96. 2,3,4,7,8-Pentachlorodibenzofuran .		1613B[10].					
97. 1,2,3,7,8,-Pentachlorodibenzo-*p*-dioxin.		1613B[10].					
98. Pentachlorophenol	604	625, 1625B		6410 B, 6630 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 140; See footnote[9], p. 27
99. Phenanthrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
100. Phenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote[9], p. 27
101. Pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
102. 2,3,7,8-Tetrachlorodibenzofuran.		1613B10.					
103. 2,3,7,8-Tetrachlorodibenzo-p-dioxin.		613, 625[5a], 1613B[10].					
104. 1,1,2,2-Tetra-chloro ethane .	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		See footnote[3], p. 130
105. Tetrachloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		See footnote[3], p. 130
106. Toluene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97.		
107. 1,2,4-Trichlorobenzene.	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote[3], p. 130; See footnote[9], p. 27

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number [2, 7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
108. 1,1,1-Trichloro-ethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
109. 1,1,2-Trichloro-ethane	601	624, 1624B	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C–97		See footnote [3], p. 130.	
110. Trichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
111. Trichlorofluoro-methane.	601	624		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
112. 2,4,6-Trichlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote [9], p. 27
113. Vinyl chloride	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], ≤6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		

[1] All parameters are expressed in micrograms per liter (µg/L) except for Method 1613B in which the parameters are expressed in picograms per liter (pg/L).

[2] The full text of Methods 601–613, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The full text of Method 1613B is incorporated by reference into this Part 136 and is available from the National Technical Information Services as stock number PB95–104774. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

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

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

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

[5a] 625, screening only.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the *Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater* (1981).

[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 (See Appendix A of this Part 136) in accordance with procedures each in Section 8.2 of each of these methods. Additionally, each laboratory, on an 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" 3M Corporation Revised 10/28/94.

[9] USGS Method 0–3116–87 from "Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments," U.S. Geological Survey, Open File Report 93–125.

[10] 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.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]

Parameter	Method	EPA [2, 7]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
1. Aldrin	GC	608	6630 B & C		D3086–90, D5812-96 (2002)	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
	GC/MS	625	6410 B	6410 B–00.		
2. Ametryn	GC					See footnote [3], p. 83; See footnote [6], p S68
3. Aminocarb	TLC					See footnote [3], p. 94; See footnote [6], p. S16
4. Atraton	GC					See footnote [3], p. 83; See footnote [6], p. S68
5. Atrazine	GC					See footnote [3], p. 83; See footnote [6], p. S68; See footnote [9]
6. Azinphos methyl	GC					See footnote [3], p. 25; See footnote [6], p. S51
7. Barban	TLC					See footnote [3], p. 104; See footnote [6], p. S64
8. α–BHC	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote [3], p. 7; See footnote [8]
	GC/MS	625 [5]	6410 B	6410 B–00.		
9. β–BHC	GC	608	6630 C		D3086–90, D5812–96(02)	See footnote [8]
	GC/MS	625 [5]	6410 B	6410 B–00.		

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES[1]—Continued

Parameter	Method	EPA[2, 7]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
10. δ–BHC	GC	608	6630 C		D3086–90, D5812–96(02)	See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
11. γ-BHC (Lindane)	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
12. Captan	GC		6630 B		D3086–90, D5812–96(02)	See footnote[3], p. 7
13. Carbaryl	TLC					See footnote[3], p. 94, See footnote[6], p. S60
14. Carbo-phenothion	GC					See footnote[4], p. 27; See footnote[6], p. S73
15. Chlordane	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
16. Chloro-propham	TLC					See footnote[3], p. 104; See footnote[6], p. S64.
17. 2,4-D	GC		6640 B			See footnote[3], p. 115; See footnote[4], p. 40
18. 4,4′-DDD	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
19. 4,4′-DDE	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
20. 4,4′-DDT	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
21. Demeton-O	GC					See footnote[3], p. 25; See footnote[6], p. S51
22. Demeton-S	GC					See footnote[3], p. 25; See footnote[6], p. S51
23. Diazinon	GC					See footnote[3], p. 25; See footnote[4], p. 27; See footnote[6], p. S51
24. Dicamba	GC					See footnote[3], p. 115
25. Dichlofen-thion	GC					See footnote[4], p. 27; See footnote[6], p. S73
26. Dichloran	GC		6630 B & C			See footnote[3], p. 7
27. Dicofol	GC				D3086–90, D5812–96(02).	
28. Dieldrin	GC	608	6630 B & C			See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
29. Dioxathion	GC					See footnote[4], p. 27; See footnote[6], p. S73
30. Disulfoton	GC					See footnote[3], p. 25; See footnote[6], p. S51
31. Diuron	TLC					See footnote[3], p. 104; See footnote[6], p. S64
32. Endosulfan I	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
33. Endosulfan II	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
34. Endosulfan Sulfate	GC	608	6630 C			See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
35. Endrin	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
36. Endrin aldehyde	GC	608				See footnote[8]
	GC/MS	625				
37. Ethion	GC					See footnote[4], p. 27; See footnote[6], p. S73
38. Fenuron	TLC					See footnote[3], p. 104; See footnote[6], p. S64
39. Fenuron-TCA	TLC					See footnote[3], p. 104; See footnote[6], p. S64
40. Heptachlor	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
41. Heptachlor epoxide	GC	608	6630 B & C		D3086–90, D5812– 96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[6], p. S73; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
42. Isodrin	GC					See footnote[4], p. 27; See footnote[6], p. S73
43. Linuron	GC					See footnote[3], p. 104; See footnote[6], p. S64
44. Malathion	GC		6630 C			See footnote[3], p. 25; See footnote[4], p. 27; See footnote[6], p. S51

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES[1]—Continued

Parameter	Method	EPA[2, 7]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
45. Methiocarb	TLC					See footnote[3], p. 94; See footnote[6], p. S60
46. Methoxy-chlor	GC		6630 B & C		D3086–90, D5812–96(02).	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
47. Mexacar-bate	TLC					See footnote[3], p. 94; See footnote[6], p.S60
48. Mirex	GC		6630 B & C			See footnote[3], p. 7; See footnote[4], p. 27
49. Monuron	TLC					See footnote[3], p. 104; See footnote[6], p. S64
50. Monuron-TCA	TLC					See footnote[3], p. 104; See footnote[6], p. S64
51. Nuburon	TLC					See footnote[3], p. 104; See footnote[6], p. S64
52. Parathion methyl	GC		6630 C			See footnote[3], p. 25; See footnote[4], p. 27
53. Parathion ethyl	GC		6630 C			See footnote[3], p. 25; See footnote[4], p. 27
54. PCNB	GC		6630 B & C			See footnote[3], p. 7
55. Perthane	GC				D3086–90, D5812–96(02).	See footnote[4], p. 27
56. Prometon	GC					See footnote[3], p. 83; See footnote[6], p. S68; See footnote[9]
57. Prometryn	GC					See footnote[3], p. 83; See footnote[6], p. S68; See footnote[9]
58. Propazine	GC					See footnote[3], p. 83; See footnote[6], p. S68; See footnote[9]
59. Propham	TLC					See footnote[3], p. 104; See footnote[6], p. S64
60. Propoxur	TLC					See footnote[3], p. 94; See footnote[6], p. S60
61. Secbumeton	TLC					See footnote[3], p. 83; See footnote[6], p. S68
62. Siduron	TLC					See footnote[3], p. 104; See footnote[6], p. S64
63. Simazine	GC					See footnote[3], p. 83; See footnote[6], p. S68; See footnote[9]
64. Strobane	GC		6630 B & C			See footnote[3], p. 7
65. Swep	TLC					See footnote[3], p. 104; See footnote[6], p. S64
66. 2,4,5–T	GC		6640 B			See footnote[3], p. 115; See footnote[4], p. 40
67. 2,4,5–TP (Silvex)	GC		6640 B			See footnote[3], p. 115; See footnote[4], p. 40
68. Terbuthylazine	GC					See footnote[3], p. 83; See footnote[6], p. S68
69. Toxaphene	GC	608	6630 B & C		D3086–90, D5812–96(02).	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
70. Trifluralin	GC		6630 B			See footnote[3], p. 7; See footnote[9]

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

[2] The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

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

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

[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 Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

[8] "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk", 3M Corporation, Revised 10/28/94.

[9] USGS Method 0–3106–93 from "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors" U.S. Geological Survey Open File Report 94–37.

TABLE IE.—LIST OF APPROVED RADIOLOGIC TEST TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)				
		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]

TABLE IE.—LIST OF APPROVED RADIOLOGIC TEST TEST PROCEDURES—Continued

Parameter and units	Method	Reference (method number or page)				
		EPA[1]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	USGS[2]
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) Ra, 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."

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TABLE IG.—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS

EPA Survey Code	Pesticide name	CAS No.	EPA Analytical Method No.(s)
8	Triadimefon	43121–43–3	507/633/525.1/1656
12	Dichlorvos	62–73–7	1657/507/622/525.1
16	2,4–D; 2,4–D Salts and Esters [2,4–Dichloro-phenoxyacetic acid]	94–75–7	1658/515.1/615/515.2/555
17	2,4–DB; 2,4–DB Salts and Esters [2,4–Dichlorophenoxybutyric acid]	94–82–6	1658/515.1/615/515.2/555
22	Mevinphos	7786–34–7	1657/507/622/525.1
25	Cyanazine	21725–46–2	629/507
26	Propachlor	1918–16–7	1656/508/608.1/525.1
27	MCPA; MCPA Salts and Esters [2–Methyl-4-chlorophenoxyacetic acid]	94–74–6	1658/615/555
30	Dichlorprop; Dichlorprop Salts and Esters [2–(2,4–Dichlorophenoxy) propionic acid]	120–36–5	1658/515.1/615/515.2/555
31	MCPP; MCPP Salts and Esters [2–(2–Methyl-4-chlorophenoxy) propionic acid]	93–65–2	1658/615/555
35	TCMTB [2–(Thiocyanomethylthio) benzo-thiazole]	21564–17–0	637
39	Pronamide	23950–58–5	525.1/507/633.1
41	Propanil	709–98–8	632.1/1656
45	Metribuzin	21087–64–9	507/633/525.1/1656
52	Acephate	30560–19–1	1656/1657
53	Acifluorfen	50594–66–6	515.1/515.2/555
54	Alachlor	15972–60–8	505/507/645/525.1/1656
55	Aldicarb	116–06–3	531.1
58	Ametryn	834–12–8	507/619/525.1
60	Atrazine	1912–24–9	505/507/619/525.1/1656
62	Benomyl	17804–35–2	631
68	Bromacil; Bromacil Salts and Esters	314–40–9	507/633/525.1/1656
69	Bromoxynil	1689–84–5	1625/1661
69	Bromoxynil octanoate	1689–99–2	1656
70	Butachlor	23184–66–9	507/645/525.1/1656
73	Captafol	2425–06–1	1656
75	Carbaryl [Sevin]	63–25–2	531.1/632/553
76	Carbofuran	1563–66–2	531.1/632
80	Chloroneb	2675–77–6	1656/508/608.1/525.1
82	Chlorothalonil	1897–45–6	508/608.2/525.1/1656
84	Stirofos	961–11–5	1657/507/622/525.1
86	Chlorpyrifos	2921–88–2	1657/508/622
90	Fenvalerate	51630–58–1	1660
103	Diazinon	333–41–5	1657/507/614/622/525.1
107	Parathion methyl	298–00–0	1657/614/622
110	DCPA [Dimethyl 2,3,5,6-tetrachloro-terephthalate]	1861–32–1	508/608.2/525.1/515.1/515.2/1656
112	Dinoseb	88–85–7	1658/515.1/615/515.2/555
113	Dioxathion	78–34–2	1657/614.1
118	Nabonate [Disodium cyanodithio-imidocarbonate]	138–93–2	630.1
119	Diuron	330–54–1	632/553
123	Endothall	145–73–3	548/548.1
124	Endrin	72–20–8	1656/505/508/608/617/525.1
125	Ethalfluralin	55283–68–6	1656/627 See footnote 1
126	Ethion	563–12–2	1657/614/614.1
127	Ethoprop	13194–48–4	1657/507/622/525.1
132	Fenarimol	60168–88–9	507/633.1/525.1/1656
133	Fenthion	55–38–9	1657/622
138	Glyphosate [N(Phosphonomethyl) glycine]	1071–83–6	547
140	Heptachlor	76–44–8	1656/505/508/608/617/525.1
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

TABLE IG.—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS—Continued

EPA Survey Code	Pesticide name	CAS No.	EPA Analytical Method No.(s)
158	Methoxychlor	72–43–5	1656/505/508/608.2/617/525.1
172	Nabam	142–59–6	630/630.1
173	Naled	300–76–5	1657/622
175	Norflurazon	27314–13–2	507/645/525.1/1656
178	Benfluralin	1861–40–1	11656/1627
182	Fensulfothion	115–90–2	1657/622
183	Disulfoton	298–04–4	1657/507/614/622/525.1
185	Phosmet	732–11–6	1657/622.1
186	Azinphos Methyl	86–50–0	1657/614/622
192	Organo-tin pesticides	12379–54–3	Ind-01/200.7/200.9
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	625/1625/515.2/555/515.1/ 525.1
208	Permethrin	52645–53–1	608.2/508/525.1/1656/1660
212	Phorate	298–02–2	1657/622
218	Busan 85 [Potassium dimethyldithiocarbamate]	128–03–0	630/630.1
219	Busan 40 [Potassium N-hydroxymethyl-N-methyldithiocarbamate]	51026–28–9	630/630.1
220	KN Methyl [Potassium N-methyl-dithiocarbamate]	137–41–7	630/630.1
223	Prometon	1610–18–0	507/619/525.1
224	Prometryn	7287–19–6	507/619/525.1
226	Propazine	139–40–2	507/619/525.1/1656
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	505/507/619/525.1/1656
241	Carbam-S [Sodium dimethyldithiocarbanate]	128–04–1	630/630.1
243	Vapam [Sodium methyldithiocarbamate]	137–42–8	630/630.1
252	Tebuthiuron	34014–18–1	507/525.1
254	Terbacil	5902–51–2	507/633/525.1/1656
255	Terbufos	13071–79–9	1657/507/614.1/525.1
256	Terbuthylazine	5915–41–3	619/1656
257	Terbutryn	886–50–0	507/619/525.1
259	Dazomet	533–74–4	630/630.1/1659
262	Toxaphene	8001–35–2	1656/505/508/608/617/525.1
263	Merphos [Tributyl phosphorotrithioate]	150–50–5	1657/507/525.1/622
264	Trifluralin	1582–09–8	1656/508/617/627/525.1
268	Ziram [Zinc dimethyldithiocarbamate]	137–30–4	630/630.1

[1] Monitor and report as total Trifluralin.

(b) * * *

References, Sources, Costs, and Table Citations

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(6) American Public Health Association. 1992, 1995, and 1998. *Standard Methods for the Examination of Water and Wastewater.* 18th, 19th, and 20th Edition (respectively). Available from: American Public Health Association, 1015 15th Street, NW., Washington, DC 20005. Standard Methods Online are available through the Standard Methods Web site (*http://www.standardmethods.org*). Tables IA, IB, IC, ID, IE.

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(10) ASTM International. Annual Book of ASTM Standards, Water, and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994, 1996, 1999, Volume 11.02, 2000, and individual standards published after 2000. Available from: ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959, or *http://www.astm.org.* Tables IA, IB, IC, ID, and IE.

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(17) AOAC-International. Official Methods of Analysis of AOAC-International, 16th Edition, (1995). Available from: AOAC-International, 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877. Table IB, See footnote 3.

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(63) Waters Corporation. Method D6508, Rev. 2, ''Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte,'' available from Waters Corp, 34 Maple Street, Milford, MA 01757, Telephone: 508/482–2131, Fax: 508/482–3625, Table IB, See footnote 54.

(64) Kelada-01, ''Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate,'' EPA 821–B–01–009 Revision 1.2, August 2001 is available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. Telephone: 800–553–6847. Table IB, See footnote 55.

(65) 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 is available from Lachat Instruments 6645 W. Mill Road, Milwaukee, WI 53218, Telephone: 414–358–4200. Table IB, See footnote 56.

(66) ''Methods for the Determination of Metals in Environmental Samples,'' Supplement I, National Exposure Risk Laboratory-Cincinnati (NERL–CI), EPA/600/R–94/111, May 1994; and ''Methods for the Determination of Inorganic Substances in Environmental Samples,'' NERL–CI, EPA/600/R–93/100, August, 1993 are available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Telephone: 800–553–6847. Table IB.

(67) ''Determination of Inorganic Ions in Drinking Water by Ion Chromatography,'' Rev. 1.0, 1997 is available from from *http://www.epa.gov/safetwater/methods/met300.pdf.* Table IB.

(68) Table IG Methods are available in ''Methods For The Determination of Nonconventional Pesticides In

Municipal and Industrial Wastewater, Volume I,'' EPA 821–R–93–010A, August 1993 Revision I, and ''Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II,'' EPA 821–R–93–010B (August 1993) are available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Telephone: 800–553–6847.

(69) Method 245.7, Rev. 2.0, ''Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry,'' February 2005, EPA–821–R–05–001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304, Telephone: 703–461–8056. Table IB, See footnote 59.

(c) Under certain circumstances, the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon recommendation of the Alternate Test Procedure Program Coordinator, Washington, DC.

(d) Under certain circumstances, the Administrator may approve additional alternate test procedures for nationwide use, upon recommendation by the Alternate Test Procedure Program Coordinator, Washington, DC.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters are cited in Tables IA, IB, IC, ID, IE, IF, and IG are prescribed in Table II. Information in the table takes precedence over information in specific methods or elsewhere. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by the Regional Administrator, to the Alternate Test Procedure Program Coordinator, Washington, DC, for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Alternate Test Procedure Program Coordinator, the Regional Administrator may grant a variance applicable to the specific discharge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter number/name	Container[1]	Preservation[2,3]	Maximum holding time[4]
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5].	6 hours
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5].	6 hours
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5].	6 hours
Table IA—Protozoan Tests:			
8. *Cryptosporidium*	LDPE; field filtration	0–8 °C	96 hours[21]
9. *Giardia*	LDPE; field filtration	0–8 °C	96 hours[21]
Table IA—Aquatic Toxicity Tests:			
10–13. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C[16]	36 hours
Table IB—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
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months
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)	P, FP, G	Cool, ≤6 °C[18], NaOH to pH>12[6], reducing agent[5].	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO_3 or H_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
Table IB—Metals:[7]			
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

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter number/name	Container[1]	Preservation[2,3]	Maximum holding time[4]
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
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, ≤6 °C[18]	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
47. 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	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 IC—Organic Tests[8]			
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
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]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], pH to 4-5[10].	14 days[10]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols[11]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5].	7 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]
14, 17, 48, 50–52. Phthalate esters[11]	G, FP-lined cap	Cool, ≤6 °C[18]	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
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

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter number/name	Container[1]	Preservation[2,3]	Maximum holding time[4]
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
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
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
60-62, 66-72, 85, 86, 95-97, 102, 103. CDDs/CDFs[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 Preservation	G	Cool, ≤6 °C[18]	7 days
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
Table ID—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 IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH<2	6 months

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

[2] Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II 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 II 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 an aliquot split from a composite sample ; otherwise, preserve the grab sample, composite sample, or aliquot split from a composite sample with in 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (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 (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). 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; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), 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 § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

[5] Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($Na_2S_2O_3$), ascorbic acid, sodium arsenite ($NaAsO_2$), or sodium borohydride ($NaBH_4$). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1—0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If $NaBH_4$ or $NaAsO_2$ is used, 25 mg/L $NaBH_4$ or 100 mg/L $NaAsO_2$ will reduce more than 50 mg/L of chlorine (see method (Kelada-01” and/or Standard Method 4500-CN− for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500-Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

[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.

(1) Sulfur: To remove elemental sulfur (S_8), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to >12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

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

(3) 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.

(4) 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.

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

(6) 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 (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), 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] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that 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.

[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 "≤ °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.

■ 8. Section 136.4 is amended by revising the first sentence of paragraph (d) introductory text to read as follows:

§ 136.4 Application for alternate test procedures.

* * * * *

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Alternate Test Procedure Program Coordinator, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460. * * *

* * * * *

■ 9. Section 136.5 is amended as follows:

■ a. In paragraph (b) by revising the last sentence.

■ b. By revising paragraph (c).

■ c. In paragraph (d) by revising the second and third sentences.

■ d. By revising paragraphs (e)(1) and (e)(2).

§ 136.5 Approval of alternate test procedures.

* * * * *

(b) * * * Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application and shall forward this decision to the Director of the State Permit Program and to the Alternate Test Procedure Program Coordinator, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

(c) Before approving any application for an alternate test procedure proposed by the responsible person or firm making the discharge, the Regional Administrator shall forward a copy of the application to the Alternate Test Procedure Program Coordinator, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

(d) * * * Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Alternate Test Procedure Program Coordinator, Washington, DC. A copy of all approval and rejection notifications will be forwarded to the Alternate Test Procedure Program Coordinator, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460, for the purposes of national coordination.

(e) *Approval for nationwide use.* (1) As expeditiously as is practicable after receipt by the Alternate Test Procedure Program Coordinator, Washington, DC, of an application for an alternate test procedure for nationwide use, the Alternate Test Procedure Program Coordinator, Washington, DC, shall notify the applicant in writing whether the application is complete. If the application is incomplete, the applicant shall be informed of the information necessary to make the application complete.

(2) As expeditiously as is practicable after receipt of a complete package, the Alternate Test Procedure Program Coordinator shall perform any analysis necessary to determine whether the alternate test procedure satisfies the applicable requirements of this part, and the Alternate Test Procedure Program Coordinator shall recommend to the Administrator that he/she approve or reject the application and shall also notify the application of the recommendation.

* * * * *

■ 10. Section 136.6 is added to Part 136 to read as follows:

§ 136.6 Method Modifications and Analytical Requirements.

(a) Definitions of terms used in this Section.

(1) *Analyst* means the person or laboratory using a test procedure (analytical method) in this Part.

(2) *Chemistry of the Method* means the reagents and reactions used in a test procedure that allow determination of the analyte(s) of interest in an environmental sample.

(3) *Determinative Technique* means the way in which an analyte is identified and quantified (*e.g.*, colorimetry, mass spectrometry).

(4) *Equivalent Performance* means that the modified method produces results that meet the QC acceptance

criteria of the approved method at this part.

(5) *Method-defined Analyte* means an analyte defined solely by the method used to determine the analyte. Such an analyte may be a physical parameter, a parameter that is not a specific chemical, or a parameter that may be comprised of a number of substances. Examples of such analytes include temperature, oil and grease, total suspended solids, total phenolics, turbidity, chemical oxygen demand, and biochemical oxygen demand.

(6) *QC* means ''quality control.''

(b) *Method Modifications.*

(1) *Allowable Changes.* Except as set forth in paragraph (b)(3) of this section, an analyst may modify an approved test procedure (analytical method) provided that the chemistry of the method or the determinative technique is not changed, and provided that the requirements of paragraph (b)(2) of this section are met.

(i) Potentially acceptable modifications regardless of current method performance include changes between automated and manual discrete instrumentation; changes in the calibration range (provided that the modified range covers any relevant regulatory limit); changes in equipment such as using similar equipment from a vendor other than that mentioned in the method (*e.g.*, a purge-and-trap device from OIA rather than Tekmar), changes in equipment operating parameters such as changing the monitoring wavelength of a colorimeter or modifying the temperature program for a specific GC column; changes to chromatographic columns (treated in greater detail in paragraph (d) of this section); and increases in purge-and-trap sample volumes (provided specifications in paragraph (e) of this section are met). The changes are only allowed provided that all the requirements of paragraph (b)(2) of this section are met.

(ii) If the characteristics of a wastewater matrix prevent efficient recovery of organic pollutants and prevent the method from meeting QC requirements, the analyst may attempt to resolve the issue by using salts as specified in *Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring* (EPA 821–B–93–001, June 1993), provided that such salts do not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such salts) and that all requirements of paragraph (b)(2) of this section are met. Chlorinated samples must be dechlorinated prior to the addition of such salts.

(iii) If the characteristics of a wastewater matrix result in poor sample dispersion or reagent deposition on equipment and prevents the analyst from meeting QC requirements, the analysts may attempt to resolve the issue by adding an inert surfactant (*i.e.* a surfactant that will not affect the chemistry of the method), which may include Brij-35 or sodium dodecyl sulfate (SDS), provided that such surfactant does not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such surfactant) and that all requirements of paragraph (b)(2) of this section are met. Chlorinated samples must be dechlorinated prior to the addition of such surfactant.

(2) *Requirements.* A modified method must produce equivalent performance to the approved methods for the analyte(s) of interest, and the equivalent performance must be documented.

(i) *Requirements for Establishing Equivalent Performance*

(A) If the approved method contains QC tests and QC acceptance criteria, the modified method must use these QC tests and the modified method must meet the QC acceptance criteria. The Analyst may only rely on QC tests and QC acceptance criteria in a method if it includes wastewater matrix QC tests and QC acceptance criteria (e.g., as matrix spikes) and both initial (start-up) and ongoing QC tests and QC acceptance criteria.

(B) If the approved method does not contain QC tests and QC acceptance criteria, or if the QC tests and QC acceptance criteria in the method do not meet the requirements of paragraph (b)(2)(i)(A) of this section, the analyst must employ QC tests specified in *Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA–821–B–98–002, March 1999) and meet the QC provisions specified therein. In addition, the Analyst must perform on-going QC tests, including assessment of performance of the modified method on the sample matrix (*e.g.*, analysis of a matrix spike/matrix spike duplicate pair for every twenty samples of a discharge analyzed), and analysis of an ongoing precision and recovery sample and a blank with each batch of 20 or fewer samples.

(C) Calibration must be performed using the modified method and the modified method must be tested with every wastewater matrix to which it will be applied (up to nine distinct matrices; as described in the ATP Protocol, after validation in nine distinct matrices, the method may be applied to all wastewater matrices), in addition to any and all reagent water tests. If the performance in the wastewater matrix or reagent water does not meet the QC acceptance criteria the method modification may not be used.

(D) Analysts must test representative effluents with the modified method, and demonstrate that the results are equivalent or superior to results with the unmodified method.

(ii) *Requirements for Documentation.* The modified method must be documented in a method write-up or an addendum that describes the modification(s) to the approved method. The write-up or addendum must include a reference number (*e.g.*, method number), revision number, and revision date so that it may be referenced accurately. In addition, the organization that uses the modified method must document the results of QC tests and keep these records, along with a copy of the method write-up or addendum, for review by an auditor.

(3) *Restrictions.* An analyst may not modify an approved analytical method for a method-defined analyte. In addition, an analyst may not modify an approved method if the modification would result in measurement of a different form or species of an analyte (*e.g.*, a change to a metals digestion or total cyanide distillation). An analyst may also may not modify any sample preservation and/or holding time requirements of an approved method.

(c) *Analytical Requirements for Multi-analyte Methods (Target Analytes).* For the purpose of NPDES reporting, the discharger or permittee must meet QC requirements only for the analyte(s) being measured and reported under the NPDES permit.

(d) The following modifications to approved methods are authorized in the circumstances described below:

(1) *Capillary Column.* Use of a capillary (open tubular) GC column rather than a packed column is allowed with EPA Methods 601–613, 624, 625, and 1624B in Appendix A to this part, provided that all QC tests for the approved method are performed and all QC acceptance criteria are met. When changing from a packed column to a capillary column, retention times will change. Analysts are not required to meet retention time specified in the approved method when this change is made. Instead, analysts must generate new retention time tables with capillary columns to be kept on file along with

other startup test and ongoing QC data, for review by auditors.

(2) *Increased sample volume in purge and trap methodology.* Use of increased sample volumes, up to a maximum of 25 mL, is allowed for an approved method, provided that the height of the water column in the purge vessel is at least 5 cm. The analyst should also use one or more surrogate analytes that are chemically similar to the analytes of interest in order to demonstrate that the increased sample volume does not adversely affect the analytical results.

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■ 11. The authority citation for part 141 continues to read as follows:

Authority: 42 U.S.C. 300f, 300g–1, 300g–2, 300g–3, 300g–4, 300g–5, 300g–6, 300j–4, 300j–9, and 300j–11.

■ 12. Section 141.21 is amended by adding four sentences to the end of footnote 1 to the Table in paragraph (f)(3) to read as follows:

§ 141.21 Coliform sampling.

* * * * *

(f) * * *

(3) * * *

[1] * * * In addition, the following online versions may also be used: 9221 A, B, D–99, 9222 A, B, C–97, and 9223 B–97. Standard Methods Online are available at *http://www.standardmethods.org.* The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only Online versions that may be used.

* * * * *

■ 13. Section 141.23 is amended as follows:

■ a. In paragraph (a)(4)(i) by revising the table entries for ''Cyanide,'' ''Nitrate,'' and ''Nitrite''.

■ b. In paragraph (k)(1) by revising the table.

§ 141.23 Inorganic chemical sampling and analytical requirements.

* * * * *

(a) * * *

(4) * * *

(i) * * *

DETECTION LIMITS FOR INORGANIC CONTAMINANTS

Contaminant	MCL (mg/L)	Methodology	Detection Limit (mg/L)
*	*	* * * *	*
Cyanide	0.2	Distillation, Spectrophotometric[3]	0.02
		Distillation, Automated, Spectrophotometric[3]	0.005
		Distillation, Amenable, Spectrophotometric[4]	0.02
		Distillation, Selective Electrode[3, 4]	0.05
		UV, Distillation, Spectrophotometric[9]	0.0005
		Micro Distillation, Flow Injection, Spectrophotometric[3]	0.0006
		Ligand Exchange with Amperometry[4]	0.0005
*	*	* * * *	*
Nitrate	10 (as N)	Manual Cadmium Reduction	0.01
		Automated Hydrazine Reduction	0.01
		Automated Cadmium Reduction	0.05
		Ion Selective Electrode	1
		Ion Chromatography	0.01
		Capillary Ion Electrophoresis	0.076
Nitrite	1 (as N)	Spectrophotometric	0.01
		Automated Cadmium Reduction	0.05
		Manual Cadmium Reduction	0.01
		Ion Chromatography	0.004
		Capillary Ion Electrophoresis	0.103
*	*	* * * *	*

* * * * *

[3] Screening method for total cyanides.

[4] Measures ''free'' cyanides when distillation, digestion, or ligand exchange is omitted.

* * * * *

[9] Measures total cyanides when UV-digestor is used, and ''free'' cyanides when UV-digestor is bypassed.

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(k) * * *

(1) * * *

Contaminant	Methodology[13]	EPA	ASTM[3]	SM[4] (18th, 19th ed.)	SM[4] (20th ed.)	SM Online[22]	Other
1. Alkalinity	Titrimetric		D1067–92, 02 B	2320 B	2320 B	2320 B–97	
	Electrometric titration					I–1030–85[5].	
2. Antimony	Inductively Coupled Plasma (ICP)—Mass Spectrometry.	200.8[2]					
	Hydride-Atomic Absorption.		D3697–92, 02.				
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.			3113 B		3113 B–99	
3. Arsenic[14]	Inductively Coupled Plasma[15].	200.7[2]		3120 B	3120 B	3120 B–99.	
	ICP-Mass Spectrometry	200.8[2]					

Contaminant	Methodology[13]	EPA	ASTM[3]	SM[4] (18th, 19th ed.)	SM[4] (20th ed.)	SM Online[22]	Other
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.		D2972–97, 03 C	3113 B		3113 B–99.	
	Hydride Atomic Absorption.		D2972–97, 03 B	3114 B		3114 B–97.	
4. Asbestos	Transmission Electron Microscopy.	100.1[9]					
	Transmission Electron Microscopy.	100.2[10]					
5. Barium	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Direct			3111D		3111 D–99.	
	Atomic Absorption; Furnace.			3113 B		3113 B–99.	
6. Beryllium	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.		D3645–97, 03 B	3113 B		3113 B–99.	
7. Cadmium	Inductively Coupled Plasma.	200.7[2]					
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.			3113 B		3113 B–99.	
8. Calcium	EDTA titrimetric		D511–93, 03 A	3500–Ca D	3500–Ca B	3500–Ca B–97.	
	Atomic Absorption; Direct Aspiration.		D511–93, 03 B	3111 B		3111 B–99.	
	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
	Ion Chromatography		D6919–03.				
9. Chromium	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.			3113 B		3113 B–99.	
10. Copper	Atomic Absorption; Furnace.		D1688–95, 02 C	3113 B		3113 B–99.	
	Atomic Absorption; Direct Aspiration.		D1688–95, 02 A	3111 B		3111 B–99.	
	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
	ICP-Mass spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
11. Conductivity	Conductance		D1125–95 (Reapproved 1999) A.	2510 B	2510 B	2510 B–97.	
12. Cyanide	Manual Distillation followed by		D2036–98 A	4500–CN^{-} C	4500–CN^{-} C.		
	Spectrophotometric, Amenable.		D2036–98 B	4500–CN^{-} G	4500–CN^{-} G	4500–CN^{-} G–99.	
	Spectro-photometric Manual.		D2036–98 A	4500–CN^{-} E	4500–CN^{-} E	4500–CN^{-} E–99.	I–3300–85[5]
	Spectro-photometric Semi-automated.	335.4[6]					
	Selective Electrode			4500–CN^{-} F	4500–CN^{-} F	4500–CN^{-} F–99.	
	UV, Distillation, Spectrophotometric.						Kelada–01[17]
	Micro Distillation, Flow Injection, Spectrophotometric.						QuikChem 10–204–00–1–X[18]
	Ligand Exchange and Amperometry[21].		D6888–04				OIA–1677, DW[20]
13. Fluoride	Ion Chromatography	300.0[6], 300.1[19]	D4327–97, 03	4110 B	4110 B	4110 B–00.	
	Manual Distill.; Color. SPADNS.			4500–F^{-} B, D	4500–F^{-} B, D	4500–F^{-} B, D–97.	
	Manual Electrode		D1179–93, 99 B	4500–F^{-} C	4500–F^{-} C	4500–F^{-} C–97.	
	Automated Electrode						380–75WE[11]
	Automated Alizarin			4500–F^{-} E	4500–F^{-} E	4500–F^{-} E–97	129–71W[11]
	Capillary Ion Electrophoresis.						D6508, Rev. 2[23]
14. Lead	Atomic Absorption; Furnace.		D3559–96, 03 D	3113 B		3113 B–99.	
	ICP–Mass spectrometry	200.8[2]					

Contaminant	Methodology[13]	EPA	ASTM[3]	SM[4] (18th, 19th ed.)	SM[4] (20th ed.)	SM Online[22]	Other
	Atomic Absorption; Platform.	200.9[2]					
	Differential Pulse Anodic Stripping Voltametry.						Method 1001[16]
15. Magnesium	Atomic Absorption		D511–93, 03 B	3111 B		3111 B–99.	
	ICP	200.7[2]		3120 B	3120 B	3120 B–99.	
	Complexation Titrimetric Methods.		D511–93, 03 A	3500–Mg E	3500–Mg B	3500–Mg B–97.	
	Ion Chromatography		D6919–03.				
16. Mercury	Manual, Cold Vapor	245.1[2]	D3223–97, 02	3112 B		3112 B–99.	
	Automated, Cold Vapor	245.2[1]					
	ICP–Mass Spectrometry	200.8[2]					
17. Nickel	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
	ICP–Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Direct			3111 B		3111 B–99.	
	Atomic Absorption; Furnace.			3113 B		3113 B–99.	
18. Nitrate	Ion Chromatography	300.0[6] 300.1[19]	D4327–97, 03	4110 B	4110 B	4110 B–00	B–1011[8]
	Automated Cadmium Reduction.	353.2[6]	D3867–90 A	4500–NO_3^- F	4500–NO_3^- F	4500–NO_3^- F–00.	
	Ion Selective Electrode			4500–NO_3^- D	4500–NO_3^- D	4500–NO_3^- D–00.	601[7]
	Manual Cadmium Reduction.		D3867–90 B	4500–NO_3^- E	4500–NO_3^- E	4500–NO_3^- E–00.	
	Capillary Ion Electrophoresis.						D6508, Rev. 2[23]
19. Nitrite	Ion Chromatography	300.0[6] 300.1[19]	D4327–97, 03	4110 B	4110 B	4110 B–00	B–1011[8]
	Automated Cadmium Reduction.	353.2[6]	D3867–90 A	4500–NO_3^- F	4500–NO_3^- F	4500–NO_3^- F–00.	
	Manual Cadmium Reduction.		D3867–90 B	4500–NO_3^- E	4500–NO_3^- E	4500–NO_3^- E–00.	
	Spectrophotometric			4500–NO_2^- B	4500–NO_2^- B	4500–NO_2^- B–00.	
	Capillary Ion Electrophoresis.						D6508, Rev. 2[23]
20. Ortho-phosphate[12].	Colorimetric, Automated, Ascorbic Acid.	365.1[6]		4500–P F	4500–P F.		
	Colorimetric, ascorbic acid, single reagent.		D515–88 A	4500–P E	4500–P E.		
	Colorimetric Phosphomolybdate;						I–1601–85[5]
	Automated-segmented flow;						I–2601–90[5]
	Automated Discrete						I–2598–85[5]
	Ion Chromatography	300.0[6] 300.1[19]	D4327–97, 03	4110 B	4110 B	4110 B–00.	
	Capillary Ion Electrophoresis.						D6508, Rev. 2[23]
21. pH	Electrometric	150.1, 150.2[1]	D1293–95, 99	4500–H^+ B	4500–H^+ B	4500–H^+ B–00.	
22. Selenium	Hydride-Atomic Absorption.		D3859–98, 03 A	3114 B		3114 B–97.	
	ICP–Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.		D3859–98, 03 B	3113 B		3113 B–99.	
23. Silica	Colorimetric, Molybdate Blue.						I–1700–85[5]
	Automated-segmented Flow.						I–2700–85[5]
	Colorimetric		D859–94, 00.				
	Molybdosilicate			4500–Si D	4500–SiO_2 C	4500–SiO_2 C–97.	
	Heteropoly blue			4500–Si E	4500–SiO_2 D	4500–SiO_2 D–97.	
	Automated for Molybdate-reactive Silica.			4500–Si F	4500–SiO_2 E	4500–SiO_2 E–97.	
	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99.	
24. Sodium	Inductively Coupled Plasma.	200.7[2]					
	Atomic Absorption; Direct Aspiration.			3111 B		3111 B–99.	
	Ion Chromatography		D6919–03.				
25. Temperature	Thermometric			2550	2550	2550–00.	
26. Thallium	ICP–Mass Spectrometry	200.8[2]					

Contaminant	Methodology [13]	EPA	ASTM [3]	SM [4] (18th, 19th ed.)	SM [4] (20th ed.)	SM Online [22]	Other
	Atomic Absorption; Platform.	200.9 [2].					

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of the following documents listed in footnotes 1–11, 16–20, and 22–23 was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies of the documents may be obtained from the sources listed below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, EPA West, 1301 Constitution Avenue, NW., Room 3334, Washington, DC 20460 (Telephone: 202–566–2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html*.

[1] "Methods for Chemical Analysis of Water and Wastes," EPA/600/4–79/020, March 1983. Available at NTIS, PB84–128677.

[2] "Methods for the Determination of Metals in Environmental Samples—Supplement I," EPA/600/R–94/111, May 1994. Available at NTIS, PB95–125472.

[3] *Annual Book of ASTM Standards,* 1994, 1996, 1999, or 2003, Vols. 11.01 and 11.02, ASTM International; any year containing the cited version of the method may be used. The previous versions of D1688–95A, D1688–95C (copper), D3559–95D (lead), D1293–95 (pH), D1125–91A (conductivity) and D859–94 (silica) are also approved. These previous versions D1688–90A, C; D3559–90D, D1293–84, D1125–91A and D859–88, respectively are located in the *Annual Book of ASTM Standards,* 1994, Vol. 11.01. Copies may be obtained from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[4] *Standard Methods for the Examination of Water and Wastewater,* 18th edition (1992), 19th edition (1995), or 20th edition (1998). American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. The cited methods published in any of these three editions may be used, except that the versions of 3111 B, 3111 D, 3113 B and 3114 B in the 20th edition may not be used.

[5] Method I–2601–90, Methods for Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment, Open File Report 93–125, 1993; For Methods I–1030–85; I–1601–85; I–1700–85; I–2598–85; I–2700–85; and I–3300–85 See Techniques of Water Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A–1, 3rd edition., 1989; Available from Information Services, U.S. Geological Survey, Federal Center, Box 25286, Denver, CO 80225–0425.

[6] "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA/600/R–93/100, August 1993. Available at NTIS, PB94–120821.

[7] The procedure shall be done in accordance with the Technical Bulletin 601 " Standard Method of Test for Nitrate in Drinking Water," July 1994, PN 221890–001, Analytical Technology, Inc. Copies may be obtained from ATI Orion, 529 Main Street, Boston, MA 02129.

[8] Method B–1011, "Waters Test Method for Determination of Nitrite/Nitrate in Water Using Single Column Ion Chromatography," August 1987. Copies may be obtained from Waters Corporation, Technical Services Division, 34 Maple Street, Milford, MA 01757, Telephone: 508/482–2131, Fax: 508/482–3625.

[9] Method 100.1, "Analytical Method For Determination of Asbestos Fibers in Water," EPA/600/4–83/043, EPA, September 1983. Available at NTIS, PB83–260471.

[10] Method 100.2, "Determination of Asbestos Structure Over 10-µm In Length In Drinking Water," EPA/600/R–94/134, June 1994. Available at NTIS, PB94–201902.

[11] Industrial Method No. 129–71W, "Fluoride in Water and Wastewater," December 1972, and Method No. 380–75WE, "Fluoride in Water and Wastewater," February 1976, Technicon Industrial Systems. Copies may be obtained from Bran & Luebbe, 1025 Busch Parkway, Buffalo Grove, IL 60089.

[12] Unfiltered, no digestion or hydrolysis.

[13] Because MDLs reported in EPA Methods 200.7 and 200.9 were determined using a 2x preconcentration step during sample digestion, MDLs determined when samples are analyzed by direct analysis (i.e., no sample digestion) will be higher. For direct analysis of cadmium and arsenic by Method 200.7, and arsenic by Method 3120 B, sample preconcentration using pneumatic nebulization may be required to achieve lower detection limits. Preconcentration may also be required for direct analysis of antimony, lead, and thallium by Method 200.9; antimony and lead by Method 3113 B; and lead by Method D3559–90D, unless multiple in-furnace depositions are made.

[14] If ultrasonic nebulization is used in the determination of arsenic by Methods 200.7, 200.8, or SM 3120 B, the arsenic must be in the pentavalent state to provide uniform signal response. For Methods 200.7 and 3120 B, both samples and standards must be diluted in the same mixed acid matrix concentration of nitric and hydrochloric acid with the addition of 100 µL of 30% hydrogen peroxide per 100 mL of solution. For direct analysis of arsenic with Method 200.8 using ultrasonic nebulization, samples and standards must contain 1 mg/L of sodium hypochlorite.

[15] Starting January 23, 2006, analytical methods using the ICP–AES technology may not be used because the detection limits for these methods are 0.008 mg/L or higher. This restriction means that the two ICP–AES methods (EPA Method 200.7 and SM 3120 B) approved for use for the MCL of 0.05 mg/L may not be used for compliance determinations for the revised MCL of 0.010 mg/L. However, prior to January 23, 2006, systems may have compliance samples analyzed with these less sensitive methods.

[16] The description for Method Number 1001 for lead is available from Palintest, LTD, 21 Kenton Lands Road, P.O. Box 18395, Erlanger, KY 41018. Or from the Hach Company, P.O. Box 389, Loveland, CO 80539.

[17] The description for the Kelada-01 Method, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, And Thiocyanate," Revision 1.2, August 2001, EPA # 821–B–01–009 for cyanide is available from the National Technical Information Service (NTIS), PB 2001–108275, 5285 Port Royal Road, Springfield, VA 22161. 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.

[18] The description for the 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 2.1, November 30, 2000, for cyanide is available from Lachat Instruments, 6645 W. Mill Rd., Milwaukee, WI 53218. Telephone: 414–358–4200.

[19] "Methods for the Determination of Organic and Inorganic Compounds in Drinking Water," Vol. 1, EPA 815–R–00–014, August 2000. Available at NTIS, PB2000–106981.

[20] Method OIA–1677, DW "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," January 2004. EPA–821–R–04–001, Available from ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842–9010.

[21] Sulfide levels below those detected using lead acetate paper may produce positive method interferences. Test samples using a more sensitive sulfide method to determine if a sulfide interference is present, and treat samples accordingly.

[22] Standard Methods Online are available at *http://www.standardmethods.org*. The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only online versions that may be used.

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

* * * * *

■ 14. Section 141.24 is amended as follows:

■ a. By revising the introductory text of paragraph (e)(1).

■ b. By revising entries 23, 24, 26, 30, 49, and 50 in the table to paragraph (e)(1).

■ c. By adding a new footnote 5 to the table to paragraph (e)(1).

§ 141.24 Organic chemicals, sampling and analytical requirements.

* * * * *

(e) * * *

(1) The following documents are incorporated by reference. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be inspected at EPA's Drinking Water Docket, 1301 Constitution Avenue, NW., EPA West, Room 3334, Washington, DC 20460 (Telephone: 202–566–2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.* Method 508A and 515.1 are in *Methods for the Determination of Organic Compounds in Drinking Water,* EPA/600/4–88–039, December 1988, Revised, July 1991. Methods 547, 550 and 550.1 are in *Methods for the Determination of Organic Compounds in Drinking Water—Supplement I,* EPA/600–4–90–020, July 1990. Methods 548.1, 549.1, 552.1 and 555 are in *Methods for the Determination of Organic Compounds in Drinking Water—Supplement II,* EPA/600/R–92–129, August 1992. Methods 502.2, 504.1, 505, 506, 507, 508, 508.1, 515.2, 524.2 525.2, 531.1, 551.1 and 552.2 are in *Methods for the Determination of Organic Compounds in Drinking Water—Supplement III,* EPA/600/R–95–131, August 1995. Method 1613 is titled "Tetra–through Octa–Chlorinated Dioxins and Furans by Isotope–Dilution HRGC/HRMS," EPA/821–B–94–005, October 1994. These documents are available from the National Technical Information Service, NTIS PB91–231480, PB91–146027, PB92–207703, PB95–261616 and PB95–104774, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll free number is: 800–553–6847. Method 6651 shall be followed in accordance with *Standard Methods for the Examination of Water and Wastewater,* 18th edition (1992), 19th edition (1995), or 20th edition (1998), American Public Health Association (APHA); any of these three editions may be used. Method 6610 shall be followed in accordance with *Standard Methods for the Examination of Water and Wastewater,* (18th Edition Supplement) (1994), or with the 19th edition (1995) or 20th edition (1998) of *Standard Methods for the Examination of Water and Wastewater;* any of these publications may be used. The APHA documents are available from APHA, 1015 Fifteenth Street NW., Washington, DC 20005. Other required analytical test procedures germane to the conduct of these analyses are contained in *Technical Notes on Drinking Water Methods,* EPA/600/R–94–173, October 1994, NTIS PB95–104766. EPA Methods 515.3 and 549.2 are available from U.S.

Environmental Protection Agency, National Exposure Research Laboratory (NERL)–Cincinnati, 26 West Martin Luther King Drive, Cincinnati, OH 45268. ASTM Method D 5317–93, 98 (Reapproved 2003) is available in the *Annual Book of ASTM Standards,* (1999), Vol. 11.02, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, any edition containing the cited version of the method may be used. EPA Method 515.4, ''Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Microextraction, Derivatization and Fast Gas Chromatography with Electron Capture Detection,'' Revision 1.0, April 2000, EPA/815/B–00/001 and EPA Method 552.3, ''Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Microextraction, Derivatization, and Gas Chromatography with Electron Capture Detection,'' Revision 1.0, July 2003, EPA 815–B–03–002, can be accessed and downloaded directly online at *http://www.epa.gov/safewater/methods/sourcalt.html.* Syngenta Method AG–625, ''Atrazine in Drinking Water by Immunoassay,'' February 2001, is available from Syngenta Crop Protection, Inc., 410 Swing Road, P.O. Box 18300, Greensboro, NC 27419. Telephone: 336–632–6000. Method 531.2 ''Measurement of N-methylcarbamoyloximes and N-methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization,'' Revision 1.0, September 2001, EPA 815–B–01–002, can be accessed and downloaded directly online at *http://www.epa.gov/safewater/methods/sourcalt.html.*

Contaminant	EPA Method[1]	Standard Methods	ASTM	Other
* * *	*	*	*	*
23. 2,4–D[4] (as acids, salts, and esters)	515.2, 555, 515.1, 515.3, 515.4.	....	D5317–93, 98 (Reapproved 2003).	
24. 2,4,5–TP[4] (Silvex)	515.2, 555, 515.1, 515.3, 515.4.	....	D5317–93, 98 (Reapproved 2003).	
* * *	*	*	*	*
26. Atrazine[2]	507, 525.2, 508.1, 505, 551.1.	....	....	Syngenta[5] AG–625
* * *	*	*	*	*
30. Dalapon	552.1 515.1, 552.2, 515.3, 515.4, 552.3.			
* * *	*	*	*	*
49. Pentachlorophenol	515.2, 525.2, 555, 515.1, 515.3, 515.4.	....	D5317–93, 98 (Reapproved 2003).	
50. Picloram[4]	515.2, 555, 515.1, 515.3, 515.4.	....	D5317–93, 98 (Reapproved 2003).	
* * *	*	*	*	*

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[1] For previously approved EPA methods which remain available for compliance monitoring until June 1, 2001, see paragraph (e)(2) of this section.

[2] Substitution of the detector specified in Method 505, 507, 508, or 508.1 for the purpose of achieving lower detection limits is allowed as follows: Either an electron capture or nitrogen phosphorus detector may be used provided all regulatory requirements and quality control criteria are met.

* * * * *

[4] Accurate determination of the chlorinated esters requires hydrolysis of the sample as described in EPA Methods 515.1, 515.2, 515.3, 515.4, and 555 and ASTM Method D 5317–93, 98 (Reapproved 2003).

[5] This method may not be used for the analysis of atrazine in any system where chlorine dioxide is used for drinking water treatment. In samples from all other systems, any result for atrazine generated by Method AG–625 that is greater than one-half the maximum contaminant level (MCL) (in other words, greater than 0.0015mg/L or 1.5 µg/L) must be confirmed using another approved method for this contaminant and should use additional volume of the original sample collected for compliance monitoring. In instances where a result from Method AG–625 triggers such confirmatory testing, the confirmatory result is to be used to determine compliance.

* * * * *

■ 15. Section 141.25 is amended by revising the table in paragraph (a) to read as follows:

§ 141.25 Analytical methods for radioactivity.

(a) * * *

Contaminant	Methodology	Reference (Method of Page Number)								
		EPA[1]	EPA[2]	EPA[3]	EPA[4]	SM[5]	ASTM[6]	USGS[7]	DOE[8]	Other
Naturally Occurring:										
Gross alpha[11] and beta	Evaporation	900.0	p. 1 ...	00–01	p. 1	302, 7110 B, 7110 B–00.	....	R–1120–76.		
Gross alpha[11]	Coprecipitation	....	....	00–02	....	7110 C, 7110 C–00.				

Contaminant	Methodology	Reference (Method of Page Number)								
		EPA [1]	EPA [2]	EPA [3]	EPA [4]	SM [5]	ASTM [6]	USGS [7]	DOE [8]	Other
Radium 226	Radon emanation	903.1	p. 16	Ra–04	p. 19	305, 7500–Ra C, 7500–Ra C–01.	D3454–97	R–1141–76	Ra-04	NY [9],
	Radiochemical	903.0	p. 13	Ra-03	....	304, 7500–Ra B, 7500–Ra B–01.	D2460–97	R–1140–76	....	GA [14]
Radium 228	Radiochemical	904.0	p. 24	Ra-05	p. 19	7500–Ra D, 7500–Ra D–01.	....	R–1142–76	....	NY [9], NJ [10], GA [14]
Uranium [12]	Radiochemical	908.0	....	....	....	7500–U B, 7500–U B–00.				
	Fluorometric	908.1	....	....	....	7500–U C (17th Ed.).	D2907–97	R–1180–76, R–1181–76.	U–04.	
	ICP–MS	200.8 [13]	....	....	....	3125	D5673–03.			
	Alpha Spectrometry	....	....	00–07	p. 33	7500–U C (18th, 19th, or 20th Ed.), 7500–U C–00.	D3972–97, 02.	R–1182–76	U–02.	
	Laser Phosphorimetry.	....	....	....	....	....	D5174–97, 02.			
Man-Made:										
Radioactive Cesium	Radiochemical	901.0	p. 4	....	....	7500–Cs B, 7500–Cs B–00.	D2459–72	R–1111–76.		
	Gamma Ray Spectrometry.	901.1	....	....	p. 92	7120, 7120–97.	D3649–91, 98a.	R–1110–76	4.5.2.3.	
Radioactive Iodine	Radiochemical	902.0	p. 6	....	....	7500–I B, 7500–I B–00.				
			p. 9	....	....	7500–I C, 7500–I C–00.				
						7500–I D, 7500–I D–00.	D3649–91, 98a.			
	Gamma Ray Spectrometry.	901.1	....	....	p. 92	7120, 7120–97.	D4785–93, 00a.	....	4.5.2.3.	
Radioactive Strontium 89, 90.	Radiochemical	905.0	p. 29	Sr–04	p. 65	303, 7500–Sr B, 7500–Sr B–01.	....	R–1160–76	Sr–01, Sr–02.	
Tritium	Liquid Scintillation	906.0	p. 34	H–02	p. 87	306, 7500–^{3}H B, 7500–^{3}H B–00.	D4107–91, 98 (Reapproved 2002).	R–1171–76.		
Gamma Emitters	Gamma Ray Spectrometry.	901.1	....	....	p. 92	7120, 7120–97.	D3649–91, 98a.	R–1110–76	Ga–01–R.	
		902.0	....	....	....	7500–Cs B, 7500–Cs B–00.	D4785–93, 00a.			
		901.0	....	....	....	7500–I B, 7500–I B–00.				

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of documents 1 through 10 and 13 through 14 was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies of the documents may be obtained from the sources listed below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, EPA West, 1301 Constitution Avenue, NW., Room 3334 , Washington, DC 20460 (Telephone: 202–566–2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.*

[1] ''Prescribed Procedures for the Measurement of Radioactivity in Drinking Water,'' EPA 600/4–80–032, August 1980. Available at the U.S. Department of Commerce, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 (Telephone 800–553–6847), PB 80–224744.

[2] ''Interim Radiochemical Methodology for Drinking Water,'' EPA 600/4–75–008 (revised), March 1976. Available NTIS, ibid.

[3] ''Radiochemistry Procedures Manual,'' EPA 520/5–84–006, December 1987. Available NTIS, ibid.

[4] ''Radiochemical Analytical Procedures for Analysis of Environmental Samples,'' March 1979. Available at NTIS, ibid. EMSL LV 053917.

[5] ''Standard Methods for the Examination of Water and Wastewater,'' 13th, 17th, 18th, 19th or 20th edition, 1971, 1989, 1992, 1995, 1998. Available at American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. Methods 302, 303, 304, 305 and 306 are only in the 13th edition. Methods 7110B, 7500–Ra B, 7500–Ra C, 7500–Ra D, 7500–U B, 7500–Cs B, 7500–I B, 7500–I C, 7500–I D, 7500–Sr B, and 7500–^{3}H B are in the 17th, 18th, 19th and 20th editions. Method 7110 C is in the 18th, 19th and 20th editions. Method 7500–U C Fluorometric Uranium is only in the 17th Edition, and 7500–U C Alpha spectrometry is only in the 18th, 19th and 20th editions. Method 7120 is only in the 19th and 20th editions. Method 3125 is only in the 20th edition. Methods 7110 B–00, 7110 C–00, 7500–Ra B–01, 7500–Ra C–01, 7500–Ra D–01, 7500–U B–00, 7500–U C–00, 7500–I B–00, 7500–I C–00, 7500–I D–00, 7120–97, 7500–Sr B–01, and 7500–^{3}H B–00 are available online at *http://www.standardmethods.org.* The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only online versions that may be used.

[6] *Annual Book of ASTM Standards,* Vol. 11.01 and 11.02, 2002; ASTM International; any year containing the cited version of the method may be used. Copies of these two volumes and the 2003 version of D 5673–03 may be obtained from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959.

[7] ''Methods for Determination of Radioactive Substances in Water and Fluvial Sediments,'' Chapter A5 in Book 5 of *Techniques of Water-Resources Investigations of the United States Geological Survey,* 1977. Available at U.S. Geological Survey (USGS) Information Services, Box 25286, Federal Center, Denver, CO 80225–0425.

[8] ''EML Procedures Manual,'' 28th (1997) or 27th (1990) Editions, Volumes 1 and 2; either edition may be used. In the 27th Edition Method Ra-04 is listed as Ra-05 and Method Ga-01–R is listed as Sect. 4.5.2.3. Available at the Environmental Measurements Laboratory, U.S. Department of Energy (DOE), 376 Hudson Street, New York, NY 10014–3621.

[9] ''Determination of Ra–226 and Ra–228 (Ra–02),'' January 1980, Revised June 1982. Available at Radiological Sciences Institute for Laboratories and Research, New York State Department of Health, Empire State Plaza, Albany, NY 12201.

[10] ''Determination of Radium 228 in Drinking Water,'' August 1980. Available at State of New Jersey, Department of Environmental Protection, Division of Environmental Quality, Bureau of Radiation and Inorganic Analytical Services, 9 Ewing Street, Trenton, NJ 08625.

[11] Natural uranium and thorium-230 are approved as gross alpha calibration standards for gross alpha with co-precipitation and evaporation methods; americium-241 is approved with co-precipitation methods.

[12] If uranium (U) is determined by mass, a 0.67 pCi/µg of uranium conversion factor must be used. This conversion factor is based on the 1:1 activity ratio of U–234 and U–238 that is characteristic of naturally occurring uranium.

[13] ''Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry,'' Revision 5.4, which is published in ''Methods for the Determination of Metals in Environmental Samples—Supplement I,''' EPA 600–R–94–111, May 1994. Available at NTIS, PB 95–125472.

[14] "The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors," Revision 1.2, December 2004. Available from the Environmental Resources Center, Georgia Institute of Technology, 620 Cherry Street, Atlanta, GA 30332–0335, USA, Telephone: 404–894–3776. This method may be used to analyze for radium-226 and radium-228 in samples collected after January 1, 2005 to satisfy the radium-226 and radium-228 monitoring requirements specified at 40 CFR 141.26.

* * * * *

■ 16. Section 141.74 is amended as follows:

■ a. By revising the entry for turbidity in the table in paragraph (a)(1).

■ b. By revising footnotes 1, 8, 10, 11, and 12 to the table in paragraph (a)(1).

■ c. By adding footnote 13 to the table in paragraph (a)(1).

■ d. By revising paragraph (a)(2).

§ 141.74 Analytical and monitoring requirements.

(a) * * *

(1) * * *

Organism	Methodology	Citation[1]
* * *	* * *	*
Turbidity[13]	Nephelometric Method	2130 B
	Nephelometric Method	180.1[9]
	Great Lakes Instruments	Method 2[10]
	Hach FilterTrak	10133[12]

* * * * *

[1] Except where noted, all methods refer to *Standard Methods for the Examination of Water and Wastewater,* 18th edition (1992), 19th edition (1995), or 20th edition (1998), American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. The cited methods published in any of these three editions may be used. In addition, the following online versions may also be used: 2130 B–01, 9215 B–00, 9221 A, B, C, E–99, 9222 A, B, C, D–97, and 9223 B–97. Standard Methods Online are available at *http://www.standardmethods.org.* The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only Online versions that may be used.

* * * * *

[8] A–1 broth may be held up to 7 days in a tightly closed screw cap tube at 4 °C.

[9] "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA/600/R–93/100, August 1993. Available at NTIS, PB94–121811.

[10] GLI Method 2, "Turbidity," November 2, 1992, Great Lakes Instruments, Inc., 8855 North 55th Street, Milwaukee, WI 53223.

[11] A description of the SimPlate method, "IDEXX SimPlate TM HPC Test Method for Heterotrophs in Water," November 2000, can be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092, telephone (800) 321–0207.

[12] A description of the Hach FilterTrak Method 10133, "Determination of Turbidity by Laser Nephelometry," January 2000, Revision 2.0, can be obtained from; Hach Co., P.O. Box 389, Loveland, CO 80539–0389, telephone: 800–227–4224.

[13] 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.

(2) Public water systems must measure residual disinfectant concentrations with one of the analytical methods in the following table. If approved by the State, residual disinfectant concentrations for free chlorine and combined chlorine also may be measured by using DPD colorimetric test kits. In addition States may approve the use of the ITS free chlorine test strip for the determination of free chlorine. Use of the test strips is described in Method D99–003, "Free Chlorine Species (HOCl- and OCl-) by Test Strip," Revision 3.0, November 21, 2003, available from Industrial Test Systems, Inc., 1875 Langston St., Rock Hill, SC 29730. Free and total chlorine residuals may be measured continuously by adapting a specified chlorine residual method for use with a continuous monitoring instrument provided the chemistry, accuracy, and precision remain the same. Instruments used for continuous monitoring must be calibrated with a grab sample measurement at least every five days, or with a protocol approved by the State.

Residual	Methodology	SM[1]	SM Online[2]	Other
Free Chlorine	Amperometric Titration	4500–Cl D	4500–Cl D–00	D1253–03[3]
	DPD Ferrous Titrimetric	4500–Cl F	4500–Cl F–00.	
	DPD Colorimetric	4500–Cl G	4500–Cl G–00.	
	Syringaldazine (FACTS)	4500–Cl H	4500–Cl H–00.	
Total Chlorine	Amperometric Titration	4500–Cl D	4500–Cl D–00	D1253–03[3]
	Amperometric Titration (low level measurement).	4500–Cl E	4500–Cl E–00.	
	DPD Ferrous Titrimetric	4500–Cl F	4500–Cl F–00.	
	DPD Colorimetric	4500–Cl G	4500–Cl G–00.	
	Iodometric Electrode	4500–Cl I	4500–Cl I–00.	
Chlorine Dioxide	Amperometric Titration	4500–ClO_2 C	4500–ClO_2 C–00.	
	DPD Method	4500–ClO_2 D.		
	Amperometric Titration	4500–ClO_2 E	4500–ClO_2 E–00.	
	Spectrophotometric			327.0, Revision 1.1[4]
Ozone	Indigo Method	4500–O_3 B	4500–O_3 B–97.	

[1] All the listed methods are contained in the 18th, 19th, and 20th editions of *Standard Methods for the Examination of Water and Wastewater,* 1992, 1995, and 1998; the cited methods published in any of these three editions may be used.

[2] Standard Methods Online are available at *http://www.standardmethods.org.* The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only Online versions that may be used.

[3] *Annual Book of ASTM Standards,* Vol. 11.01, 2004 ; ASTM International; any year containing the cited version of the method may be used. Copies of this method may be obtained from ASTM International, 100 Barr Harbor Drive, P.O. Box C700 West Conshohocken, PA 19428–2959.

[4] EPA Method 327.0, Revision 1.1, "Determination of Chlorine Dioxide and Chlorite Ion in Drinking Water Using Lissamine Green B and Horseradish Peroxidase with Detection by Visible Spectrophotometry," USEPA, May 2005, EPA 815–R–05–008. Available online at *http://www.epa.gov/safewater/methods/sourcalt.html.*

* * * * *

PART 143—NATIONAL SECONDARY DRINKING WATER REGULATIONS

■ 17. The authority citation for part 143 continues to read as follows:

Authority: 42 U.S.C. 300f *et seq.*

■ 18. Section § 143.4 is amended by revising the table in paragraph (b) to read as follows:

§ 143.4 Monitoring.

* * * * *

(b) * * *

Contaminant	EPA	ASTM[3]	SM[4] 18th and 19th ed.	SM[4] 20th ed.	SM[7] Online	Other
1. Aluminum	200.7[2]		3120 B	3120 B	3120 B–99.	
	200.8[2]		3113 B		3113 B–99.	
	200.9[2]		3111 D		3111 D–99.	
2. Chloride	300.0[1], 300.1[6]	D4327–97, 03	4110 B	4110 B	4110 B–00.	
			4500–Cl^{-} D	4500–Cl^{-} D	4500–Cl^{-} D–97.	
		D512–89 (Reapproved 1999) B.	4500–Cl^{-} B	4500–Cl^{-} B	4500–Cl^{-} B–97.	
						D6508, Rev. 2[8]
3. Color			2120 B	2120 B	2120 B–01.	
4. Foaming Agents			5540 C	5540 C	5540 C–00.	
5. Iron	200.7[2]		3120 B	3120 B	3120 B–99.	
	200.9[2]		3111 B		3111 B–99.	
			3113 B		3113 B–99.	
6. Manganese	200.7[2]		3120 B	3120 B	3120 B–99.	
	200.8[2]		3111 B		3111 B–99.	
	200.9[2]		3113 B		3113 B–99.	
7. Odor			2150 B	2150 B	2150 B–97.	
8. Silver	200.7[2]		3120 B	3120 B	3120 B–99.	I–3720–85[5]
	200.8[2]		3111 B		3111 B–99.	
	200.9[2]		3113 B		3113 B–99.	
9. Sulfate	300.0[1], 300.1[6]	D4327–97, 03	4110 B	4110 B	4110 B–00.	
	375.2[1]		4500–SO_4^{2-} F	4500–SO_4^{2-} F.		
			4500–SO_4^{2-} C, D	4500–SO_4^{2-} C, D.		
		D516–90, 02	4500–SO_4^{2-} E	4500–SO_4^{2-} E.		
						D6508, Rev. 2[8]
10. Total Dissolved Solids			2540 C	2540 C	2540 C–97.	
11. Zinc	200.7[2]		3120 B	3120 B	3120 B–99.	
	200.8[2]		3111 B		3111 B–99.	

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of the following documents was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies of the documents may be obtained from the sources listed below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, EPA West, 1301 Constitution Avenue, NW., Room 3334, Washington, DC (Telephone: 202–566–2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.*

[1] "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA/600/R–93–100, August 1993. Available at NTIS, PB94–120821.

[2] "Methods for the Determination of Metals in Environmental Samples—Supplement I," EPA/600/R–94–111, May 1994. Available at NTIS, PB 95–125472.

[3] *Annual Book of ASTM Standards,* 1994, 1996, 1999, or 2004, Vols. 11.01 and 11.02, ASTM International; any year containing the cited version of the method may be used. Copies may be obtained from the ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[4] *Standard Methods for the Examination of Water and Wastewater,* 18th edition (1992), 19th edition (1995), or 20th edition (1998). American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. The cited methods published in any of these three editions may be used, except that the versions of 3111 B, 3111 D, and 3113 B in the 20th edition may not be used.

[5] Method I–3720–85, *Techniques of Water Resources Investigation of the U.S. Geological Survey,* Book 5, Chapter A–1, 3rd ed., 1989. Available from Information Services, U.S. Geological Survey, Federal Center, Box 25286, Denver, CO 80225–0425.

[6] "Methods for the Determination of Organic and Inorganic Compounds in Drinking Water," Vol. 1, EPA 815-R–00–014, August 2000. Available at NTIS, PB2000–106981.

[7] Standard Methods Online are available at *http://www.standardmethods.org.* The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only online versions that may be used.

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

PART 430—THE PULP, PAPER, AND PAPERBOARD POINT SOURCE CATEGORY

■ 19. The authority citation for part 430 continues to read as follows:

Authority: Sections 301, 304, 306, 307, 308, 402, and 501 of the Clean Water Act, as amended, (33 U.S.C. 1311, 1314, 1316, 1317, 1318, 1342, and 1361), and Section 112 of the Clean Air Act, as amended (42 U.S.C. 7412).

■ 20. Section 430.02 is amended by adding paragraph (g) to read as follows:

§ 430.02 Monitoring requirements.

* * * * *

(g) Analyst may use NCASI Method CP–86.07, "Chlorinated Phenolics in Water by *In situ* Acetylation and GC/MS Determination" (January 2002) for determination of certain chlorinated phenols, chlorinated guaiacols, chlorinated catechols, chlorinated benzaldehydes (i.e., vanillins and syringaldehydes), and trichlorsyringol (analytes specified in the method) in bleach plant filtrate as an alternative to EPA Method 1653. NCASI Method CP–86.07 is available from the Publications Coordinator, NCASI, P.O. Box 13318, Research Triangle Park, NC 27709–3318. Telephone: 919–588–1987.

PART 455—PESTICIDE CHEMICALS

■ 21. The authority citation for Part 455 continues to read as follows:

Authority: Secs. 301, 304, 306, 307, and 501, Pub. L. 92–500, 86 Stat. 816, Pub. L. 95–217, 91 Stat. 156, and Pub. L. 100–4 (33 U.S.C. 1311, 1314, 1316, 1317, 1361).

■ 22. Section 455.50 is revised to read as follows:

§ 455.50 Identification of test procedures.

The pesticide active ingredients to which this section applies and for which effluent limitations guidelines and standards are specified in this Part are named, together with the Chemical Abstracts Service (CAS) number (provided to assist in identifying the pesticide active ingredient only) and analytical method(s) designation(s) in Table IG at 40 CFR 136.3(a). Except as provided in 40 CFR 136.5, the discharge parameter values required under the Clean Water Act must be determined by one of the analytical methods cited and described in Table IG at 40 CFR 136.3(a). Pesticide manufacturers may not use the analytical method cited in

Table IB, Table IC, or Table ID of 40 CFR 136.3(a) to make these determinations (except where the method cited in those tables is identical to the method specified in Table IG at 40 CFR 136.3(a)). The full texts of the analytical methods cited in Table IG at 40 CFR 136.3(a) are contained in the *Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I,* EPA 821-R–93–010A (August 1993 Revision I) and *Volume II,* EPA 821-R–93–010B (August 1993) (the ''Compendium''). Each pesticide chemical manufacturer that is required to determine discharge parameter values under this Part using one of the analytical methods cited in Table IG at 40 CFR 136.3(a) must request in writing a copy of the Compendium from the permit authority or local control authority (as applicable) prior to determining such discharge parameter values, unless the manufacturer already has a copy.

■ 23. Part 455 is amended by removing and reserving Table 7 to Part 455.

PART 465—COIL COATING POINT SOURCE CATEGORY

■ 24. The authority citation for Part 465 continues to read as follows:

Authority: Secs. 301,304 (b), (c), (e), and (g), 306 (b) and (c), 307 (b) and (c), and 501 of the Clean Water Act (the Federal Water Pollution Control Act of 1972, as amended by the Clean Water Act of 1977)(the ''Act''); 33 U.S.C. 1311, 1314 (b), (c), (e), and (g), 1316 (b) and (c), 1317 (b) and (c), and 1361; 86 Stat. 816, Pub. L. 92–500; 91 Stat. 1567, Pub. L. 95–217.

■ 25. Section 465.03 is amended by revising paragraph (c) to read as follows:

§ 465.03 Monitoring and reporting requirements.

* * * * *

(c) The analytical method required for determination of petroleum hydrocarbons (non-polar material) is given under the listing for ''oil and grease'' at 40 CFR 136.3(a), Table IB and must be used after December 31, 2005.

* * * * *

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