

Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

NR 219.01 Purpose.
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Note: A number of the references cited in this chapter are no longer in print. Copies of references which are out-of-print are available at any public library by inter-library loan.

NR 219.01 Purpose. The purpose of this chapter is to establish analytical test methods, preservation procedures, requirements for laboratories, and procedures applicable to effluent limitations for discharges from point sources as authorized by ss. 299.11 and 283.55 (1), Stats.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86; am. Register, June, 1986, No. 366, eff. 7-1-86; am. Register, April, 1988, No. 388, eff. 5-1-88; corrections made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.

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

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

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

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

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86; am. (1) (intro.), Register, June, 1986, No. 366, eff. 7-1-86; correction in (1) (a) made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491; correction in (2) made under s. 13.93 (2m) (b) 7., Stats., Register October 2002 No. 562; correction in (2) made under s. 13.93 (2m) (b) 7., Stats., Register November 2004 No. 587; CR 13-112: am. (1), (2) Register May 2015 No. 713, eff. 6-1-15.

NR 219.03 Definitions. As used in this chapter:

(1) "EPA" means the U.S. environmental protection agency.

(2) "Department" means the department of natural resources.

(3) "Sludge" is defined in ss. NR 204.03 (55) and 214.03 (34).

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. (1), (2), (3) and (4m), Register, January, 1978, No. 265, eff. 2-1-78; r. and recr. Register, June, 1986, No. 366, eff. 7-1-86; r. and recr. (1), r. (3) and (4), Register, November, 1992, No. 443, eff. 12-1-92; CR 04-033: cr. (3) Register November 2004 No. 587, eff. 12-1-04.

NR 219.033 Alternate test procedures. Approvals of alternate test procedures for nationwide use and specific discharges are granted by EPA. The department may approve the use of an alternate test procedure on a case-by-case basis if the criteria for approval of the alternate procedure established in s. NR 149.42 are met. If the department or the EPA approves an alternate test procedure, it shall be considered equivalent to the approved method.

Note: The federal requirements for alternate test procedure approval are given in 40 CFR 136.5.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; r. and recr. January, 1978, No. 265, eff. 2-1-78; renum. from NR 219.04 and am. Register, June, 1986, No. 366, eff. 7-1-86; r. and recr. Register, November, 1992, No. 443, eff. 12-1-92; am. Register, February, 1996, No. 482, eff. 3-1-96; correction made under s. 13.92 (4) (b) 7., Stats., Register May 2009 No. 641, eff. 6-1-09; CR 13-112: renum. from 219.05 Register May 2015 No. 713, eff. 6-1-15.

NR 219.037 Laboratory certification or registration. Bacteriological analyses of groundwater samples, and all radiological analyses shall be performed by the state laboratory of hygiene or at a laboratory certified or approved by the department

of agriculture, trade and consumer protection. Other laboratory test results, including effluent toxicity, submitted to the department under a WPDES permit shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

- (1) Temperature,
- (2) Turbidity,
- (3) Bacteria tests in wastewater effluent and sludges,
- (4) pH,
- (5) Chlorine residual,
- (6) Specific conductance,
- (7) Physical properties of soils and sludges,
- (8) Nutrient tests of soils and sludges,
- (9) Flow measurements.

History: Cr. Register, April, 1986, No. 364, eff. 8-28-86; renum. from NR 219.07 and am. (intro.) Register, November, 1992, No. 443, eff. 7-1-93; am. Register February, 1996, No. 482, eff. 3-1-96; correction in (intro.) made under s. 13.93 (2m) (b) 6., Stats., Register November 2004 No. 587; CR 13-112: renum. from 219.06 Register May 2015 No. 713, eff. 6-1-15.

NR 219.04 Identification of test procedures.

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

(2) SAMPLE PRESERVATION PROCEDURES. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to H are prescribed in table F. Sludge samples shall be preserved at the time of collection by cooling to less than or equal to 65 C where required. All samples requiring thermal preservation at less than or equal to 65 C shall be cooled immediately after collection, and the required temperature maintained during shipping. Any person may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the regional administrator and shall provide sufficient data to assure that the variance does not adversely affect the integrity of the sample. The regional administrator will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

(3) TEMPERATURE REPORTING PROCEDURES. Samples cooled with ice packs or not in direct contact with ice during shipping shall be cooled to less than or equal to 6° C prior to shipping, and a temperature blank shall be submitted with the samples. Samples cooled during shipping with ice packs may not be recorded as received on ice. Samples may be recorded as received on ice only if solid ice is present in the cooler at the time the samples are received. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

- (a) The temperature of an actual sample.

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

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

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

Note: Copies of the publications referenced in Tables A–H are available for inspection at the offices of the department of natural resources and the legislative reference bureau. Many of these materials are also available through inter-library loan.

History: Cr. Register, June, 1986, No. 366, eff. 7–1–86; r. and recr. Tables B and E, Register, April, 1988, No. 388, eff. 5–1–88; am.; r. and recr. Tables A to F, Register,

November, 1992, No. 443, eff. 12–1–92; am. (1), am. Tables A to F, Register, April, 1994, No. 460, eff. 5–1–94; am. (1) and (2), Tables A to F, cr. (3), Register, February, 1996, No. 482, eff. 3–1–96; CR 02–019: am. Table B Register October 2002 No. 562, eff. 11–1–02; CR 04–033: r. and recr. Table A, Table B, Table BM, Table C, Table D, Table E, Table EM, and Table F, cr. Table ES Register November 2004 No. 587, eff. 12–1–04; CR 04–101: am. Table A Note 29 Register May 2005 No. 593, eff. 6–1–05; CR 08–076: am. (2) and (3) (intro.), cr. (4), r. and recr. Table A, B, C to EM and F Register May 2009 No. 641, eff. 6–1–09; correction to Table B Parameter No. 41 made under s. 13.92 (4) (b) 7., Stats., Register September 2009 No. 645; correction to Table B Parameter No. 15 made under s. 13.92 (4) (b) 7., Stats., Register April 2010 No. 652; CR 13–112: am. (1) and (2), r. and recr. Table A, B, r. Table BM, r. and recr. Table C to F, cr. Tables G and H, Register May 2015 No. 713, eff. 6–1–15; **correction in Table C footnote 11 made under s. 13.92 (4) (b) 6., Stats., Register July 2015 No. 715, eff. 8–1–15.**

Table A
List of Approved Biological Methods for Wastewater and Sewage Sludge

| Parameter and units | Analytical Technology ¹ | EPA | Standard Methods ²⁷ | AOAC, ASTM, USGS | Other |
|--|---|---|--|------------------------|---|
| Bacteria: | | | | | |
| 1. Coliform (fecal), number per 100 mL or number per gram dry weight | Most Probable Number (MPN), 5 tube, 3 dilution, or | p. 132 ³ 1680 ^{11,15} 1681 ^{11,20} | 9221 C-2006 9221 E-2006 | | |
| | Membrane filter (MF) ² , single step | p. 124 ³ | 9222 D-1997 | B-0050-85 ⁴ | |
| 2. Coliform (fecal) in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 132 ³ | 9221 C-2006 9221 E-2006 | | |
| | MF ² , single step ⁵ | p. 124 ³ | 9222 D-1997 | | |
| 3. Coliform (total), number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 ³ | 9221 B-2006 | | |
| | MF ² , single step or two step | p. 108 ³ | 9222 B-1997 | B-0025-85 ⁴ | |
| 4. Coliform (total), in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 ³ | 9221 B-2006 | | |
| | MF ² with enrichment ⁵ | p. 111 ³ | 9222 (B +B.5c)-1997 | | |
| 5. <i>E. coli</i> , number per 100 mL ²¹ | MPN ^{6,8,16} multiple tube, or | | 9221B.1-2006/ 9221F-2006 ^{12,14} | | |
| | multiple tube/multiple well, or | | 9223 B-200 ⁴ ¹³ | 991.15 ¹⁰ | Colilert [®] ^{13, 18} Colilert-18 [®] ^{13,17,18} |
| | MF ^{2,6,7,8} single step | 1603 ²² | | | mColiBlue ^{®19} |
| 6. Fecal streptococci, number per 100 mL | MPN, 5 tube 3 dilution, or | p. 139 ³ | 9230 B-2007 | | |
| | MF ² , or | p. 136 ³ | 9230 C-2007 | B-0055-85 ⁴ | |
| | Plate count | p. 143 ³ | | | |
| 7. Enterococci, number per 100 mL ²² | MPN ^{6, 8} , multiple tube/multiple well, or | | | D6503-99 ⁹ | Enterolert [®] ^{13, 24} |
| | MF ^{2, 6, 7, 8} single step or | 1600 ²⁵ | 9230 C-2007 | | |
| | Plate count | p. 143 ³ | | | |
| 8. <i>Salmonella</i> , number per gram dry weight ¹¹ | MPN multiple tube | 1682 ²³ | | | |
| Aquatic Toxicity: | | | | | |
| 9. Toxicity, acute, fresh water organisms, percent effluent | Daphnia, <i>Ceriodaphnia dubia</i> 48-h static-renewal mortality | | | | Note 27 |
| | Fathead Minnow, <i>Pimephales promelas</i> , 96-h static renewal mortality, or 96-h flow-through mortality | | | | Note 27 |
| 10. Toxicity, chronic, fresh water organisms, percent effluent | Daphnia, <i>Ceriodaphnia dubia</i> , survival and reproduction | | | | Note 27 |
| | Fathead minnow, <i>Pimephales promelas</i> , larval survival and growth | | | | Note 27 |

¹ The method must be specified when results are reported.

- ² A 0.45- μ m membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.
- ³ Microbiological Methods for Monitoring the Environment, Water, and Wastes, EPA/600/8-78/017. 1978. US EPA.
- ⁴ U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. USGS.
- ⁵ Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.
- ⁶ Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.
- ⁷ When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.
- ⁸ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.
- ⁹ Annual Book of ASTM Standards-Water and Environmental Technology, Section 11.02. 2000, 1999, 1996. ASTM International.
- ¹⁰ Official Methods of Analysis of AOAC International. 16th Edition, 4th Revision, 1998. AOAC International.
- ¹¹ Recommended for enumeration of target organism in sewage sludge.
- ¹² The multiple-tube fermentation test is used in 9221B.1-2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.
- ¹³ These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β -glucuronidase produced by *E. coli*.
- ¹⁴ After prior enrichment in a presumptive medium for total coliform using 9221B.1-2006, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h \pm 3 h of incubation shall be submitted to 9221F-2006. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 μ g/mL of MUG may be used.
- ¹⁵ Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium, EPA-821-R-10-003. April 2010. U.S. EPA.
- ¹⁶ Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert[®] may be enumerated with the multiple-well procedures, Quanti-Tray[®], Quanti-Tray[®]/2000, and the MPN calculated from the table provided by the manufacturer.
- ¹⁷ Colilert-18[®] is an optimized formulation of the Colilert[®] for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C rather than the 24 h required for the Colilert[®] test and is recommended for marine water samples.
- ¹⁸ Descriptions of the Colilert[®], Colilert-18[®], Quanti-Tray[®], and Quanti-Tray[®]/2000 may be obtained from IDEXX Laboratories, Inc. 1 IDEXX Drive, Westbrook, ME 04092.
- ¹⁹ A description of the mColiBlue24[®] test, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.
- ²⁰ Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 Medium, EPA-821-R-06-013. U.S. Environmental Protection Agency, Office of Water, Washington, DC. July 2006. U.S. EPA.
- ²¹ Recommended for enumeration of target organism in wastewater effluent.
- ²² Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC), EPA-821-R-09-007. December 2009. U.S. EPA. U.S. Environmental Protection Agency, Office of Water, Washington, DC
- ²³ Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium, EPA-821-R-06-014. July 2006. U.S. EPA.
- ²⁴ A description of the Enterolert[®] test may be obtained from IDEXX Laboratories Inc
- ²⁵ Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl- β -D-Glucoside Agar (mEI), EPA-821-R-09-016. December 2009. U.S. EPA.
- ²⁶ Standard Methods for the Analysis of Water and Wastewater. With the promulgation of Federal Register /Vol. 77, No. 97 / Friday, May 18, 2012, the EPA lists only the most recently EPA-approved version of a Standard Method (regardless of the printed or online edition) in 40 CFR Part 136, with few exceptions, to identify the method with the year of Standard Methods approval or adoption designated by the last four digits in the method number (e.g., Standard Method 3113B-2004). This approach clearly identifies the version of the standard method approved under Part 136 and no longer ties it to a particular compendium printing or edition of Standard Methods. Methods can be purchased at www.standardmethods.org/.
- ²⁷ Compliance monitoring must be performed in accordance with the specifications in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 2nd Edition," Wisconsin Department of Natural Resources, 2004. This publication is available for inspection at the offices of the Department of Natural Resources and the Legislative Reference Bureau. Copies are available from the Department of Natural Resources, Bureau of Science Services, P.O. Box 7921, Madison, WI 53707.

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|---|--|---|--------------|---|
| 1. Acidity, as CaCO ₃ , mg/L | Electrometric endpoint or phenolphthalein endpoint | | 2310 B-1997 | D1067-06 | I-1020-85 ² |
| 2. Alkalinity, as CaCO ₃ , mg/L | Electrometric or Colorimetric titration to pH 4.5, Manual | | 2320 B-1997 | D1067-06 | I-1030-85 ² 973.43 ³ |
| | Automatic | 310.2 (Rev. 1974) ¹ | | | I-2030-85 ² |
| 3. Aluminum—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 D-1999 or 3111 E-1999 | | I-3051-85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | |
| | Stabilized temperature graphite furnace AA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma-atomic emission spectrometry (ICP-AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | | I-4471-97 ⁵⁰ |
| | Inductively coupled plasma-mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14, ³ I-4471-97 ⁵⁰ |
| | Direct Current Plasma (DCP) ³⁶ | | | D4190-08 | Note ³⁴ |
| | Colorimetric (Eriochrome cyanine R) | | 3500-A1 B-2001 | | |
| 4. Ammonia (as N), mg/L | Manual distillation ⁶ or gas diffusion (pH > 11), followed by any of the following: | 350.1, Rev. 2.0 (1993) | 4500-NH ₃ B-1997 | | 973.49 ³ |
| | Titration | | 4500-NH ₃ C-1997 | | |
| | Electrode | | 4500-NH ₃ D-1997 or E-1997 | D1426-08 (B) | |
| | Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods | | 4500-NH ₃ F-1997 | | Note ⁶⁰ |
| | Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods | 350.1 ³⁰ , Rev. 2.0 (1993) | 4500-NH ₃ G-1997 4500-NH ₃ H-1997. | | I-4523-85 ² |
| | Automated electrode | | | | Note ⁷ |
| | Ion Chromatography | | | D6919-09 | |
| 5. Anti-mony—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 ⁵⁰ |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---|---|--|----------------------------------|--------------|---|
| | Inductively coupled plasma– mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| 6. Arsenic–Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | 206.5 (Issued 1978) ¹ | | | |
| | AA gaseous hydride | | 3114 B–2009 or 3114 C–2009 | D2972–08 (B) | I–3062–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D2972–08 (C) | I–4063–98 ⁴⁹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4020–05 ⁷⁰ |
| | Colorimetric (SDDC) | | 3500–As B–1997 | D2972–08 (A) | I–3060–85 ² |
| 7. Barium–Total, ⁴ mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 D–1999 | | I–3084–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D4382–02(07) | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) ³⁶ | | | | Note ³⁴ |
| 8. Beryllium–Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D–1999 or 3111 E–1999 | D3645–08 (A) | I–3095–85. ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D3645–08 (B) | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Aluminon) | | Note ⁶¹ | | |
| 9. Biochemical oxygen demand (BOD5), mg/L | Dissolved Oxygen Depletion | | 5210 B–2001 | | 973.44 ³ , p. 17 ⁹ , I–1578–78 ⁸ Notes ^{10,63} |
| 10. Boron–Total, ³⁷ mg/L | Colorimetric (Curcumin) | | 4500–B B–2000 | | I–3112–85 ² |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---|---|--|----------------------------------|--------------------------|--|
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) | | | D4190–08 | Note ³⁴ |
| 11. Bromide, mg/L | Titrimetric | | | | I–1125–85 ² |
| | Ion selective electrode (ISE) | | | D1246–05 | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) 300.1–1, Rev 1.0 (1997) | 4110 B–2000, C–2000, D–2000 | D4327–03 | 993.30 ³ |
| | CIE/UV | | 4140 B–1997 | D6508–00(05) | D6508, Rev. 2 ⁵⁴ |
| 12. Cadmium—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B–1999 or 3111 C–1999 | D3557–02(07) (A or B) | 974.27 ³ , p. 37 ⁹ , I–3135–85 ² or I–3136–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D3557–02(07) (D) | I–4138–89 ⁵¹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–1472–85 ² or I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | Voltametry ¹¹ | | | D3557–02(07) (C) | |
| | Colorimetric (Dithizone) | | 3500–Cd–D–1990 | | |
| | 13. Calcium—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | |
| AA direct aspiration (FLAA) | | | 3111 B–1999 | D511–08(B) | I–3152–85 ² |
| Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | | I–4471–97 ⁵⁰ |
| Inductively coupled plasma–mass spectrometry (ICP/MS) | | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ |
| Direct current plasma (DCP) | | | | | Note ³⁴ |
| Titrimetric (EDTA) | | | 3500–Ca B–1997 | D511–08 (A) | |
| Ion Chromatography | | | | D6919–09 | |
| 14. Carbonaceous biochemical oxygen demand (CBOD ₅), mg/L ¹² | Dissolved Oxygen Depletion with nitrification inhibitor | | 5210 B–2001 | | Note ^{35,63} |
| 15. Chemical oxygen demand (COD), mg/L | Titrimetric | 410.3 (Rev. 1978) ¹ | 5220 B–1997 or C–1997 | D1252–06 (A) | I–3560–85 ² , 973.46 ³ , p. 17 ⁹ |
| | Spectrophotometric, manual or automatic | 410.4, Rev. 2.0 (1993) | 5220 D–1997 | D1252–06 (B) | Note ^{13,14} I–3561–85 ² |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---------------------------------------|---|---|--------------------------------|---------------------|--|
| 16. Chloride, mg/L | Titrimetric: (silver nitrate) | | 4500-Cl ⁻ B-1997 | D512-04 (B) | I-1183-85 ² |
| | Colorimetric: manual | | | | I-1187-85 ² |
| | Colorimetric, Automated (Ferricyanide) | | 4500-Cl ⁻ E-1997 | | I-2187-85 ² |
| | Potentiometric Titration | | 4500-Cl ⁻ D-1997 | | |
| | Ion Selective Electrode | | | D512-04 (C) | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or 4110 C-2000 | D4327-03 | 993.30 ³ , I-2057-90 ⁵¹ |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 ⁵⁴ |
| 17. Chlorine—Total residual, mg/L | Amperometric direct | | 4500-Cl D-2000 | D1253-08 | |
| | Amperometric direct (low level) | | 4500-Cl E-2000 | | |
| | Iodometric direct | | 4500-Cl B-2000 | | |
| | Back titration ether end-point ¹⁵ | | 4500-Cl C-2000 | | |
| | Colorimetric, DPD-FAS | | 4500-Cl F-2000 | | |
| | Spectrophotometric, DPD | | 4500-Cl G-2000 | | |
| | Ion selective electrode (ISE) | | | | Note ¹⁶ |
| 17A. Chlorine—Free Available, mg/L | Amperometric direct | | 4500-Cl D-2000 | D1253-08 | |
| | Amperometric direct (low level) | | 4500-Cl E-2000 | | |
| | DPD-FAS | | 4500-Cl F-2000 | | |
| | Spectrophotometric, DPD | | 4500-Cl G-2000 | | |
| 18. Chromium VI dissolved, mg/L | 0.45-micron Filtration followed by any of the following: | | | | |
| | AA chelation-extraction | | 3111 C-1999 | | I-1232-85 ² |
| | Ion Chromatography | 218.6, Rev. 3.3 (1994) | 3500-Cr C-2009 | D5257-03 | 993.23 |
| 19. Chromium—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B-1999 | D1687-02(07) (B) | 974.27, ³ I-3236-85 ² |
| | AA chelation-extraction | | 3111 C-1999 | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|---|---|----------------------------|-------------------|---|
| 20. Cobalt—Total, ⁴ mg /L | Graphite furnace AA (GFAA) | | 3113 B–2004 | D1687–02(07) (C) | I–3233–93 ⁴⁶ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4020–05 ⁷⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Diphenyl–carbazine) | | | 3500–Cr B–2009 | |
| 21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity | AA direct aspiration (FLAA) | | 3111 B–1999 or 3111 C–1999 | D3558–08 (A or B) | p. 37, ⁹ I–3239–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D3558–08 (C) | I–4243–89 ⁵¹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev. 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4020–05 ⁷⁰ |
| | Direct current plasma (DCP) | | | D4190–08 | Note ³⁴ |
| 22. Cop- per—Total, ⁴ mg/L | Colorimetric (ADMI) | | | | Note ¹⁸ |
| | Colorimetric (Platinum cobalt) | | 2120 B–2001 | | I–1250–85 ² |
| 22. Cop- per—Total, ⁴ mg/L | AA direct aspiration (FLAA) ³⁶ | | 3111 B–1999 or 3111 C–1999 | D1688–07 (A or B) | 974.27, ³ p. 37, ⁹ I–3270–85 ² or I–3271–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D1688–07 (C) | I–4274–89 ⁵¹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev. 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4020–05 ⁷⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Neocuproine) | | | 3500–Cu B–1999 | |
| | Colorimetric (Bathocuproine) | | | 3500–Cu C–1999 | Note ¹⁹ |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|-----------------------------------|---|---|---------------------------------------|-----------------------|---|
| 23. Cyanide—Total, mg/L | Automated UV digestion/distillation and Colorimetry | | | | Kelada-01. ⁵⁵ |
| | Segmented Flow Injection, In-Line Ultraviolet Digestion, followed by gas diffusion amperometry | | | D7511-09 | |
| | Manual distillation with MgCl ₂ , followed by any of the following: | 335.4, Rev. 1.0 (1993) ⁵⁷ | 4500-CN ⁻ B-1999 or C-1999 | D2036-09(A), D7284-08 | 10-204-00-1-X ⁵⁶ |
| | Flow Injection, gas diffusion amperometry | | | D2036-09(A) D7284-08 | |
| | Titrimetric | | 4500-CN ⁻ D-1999 | D2036-09(A) | p. 22 ⁹ |
| | Colorimetry; Spectrophotometric, manual | | 4500-CN ⁻ E-1999 | D2036-09(A) | I-3300-85 ² |
| | Colorimetry; Semi-Automated ²⁰ | 335.4, Rev. 1.0 (1993) ⁵⁷ | | | 10-204-00-1-X, ⁵⁶ I-4302-85 ² |
| | Ion Chromatography | | | D2036-09(A) | |
| | Ion Selective Electrode | | 4500-CN ⁻ F-1999 | D2036-09(A) | |
| 24. Cyanide—Available, mg/L | Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl ₂ , followed by Titrimetric or Spectrophotometric | | 4500-CN ⁻ G-1999 | D2036-09(B) | |
| | Flow injection and ligand exchange, followed by gas diffusion amperometry ⁵⁹ | | | D6888-09 | OIA-1677-09 ⁴⁴ |
| | Automated Distillation and Colorimetry (no UV digestion) | | | | Kelada-01 ⁵⁵ |
| 24.A Cyanide-Free, mg/L | Flow Injection, followed by gas diffusion amperometry | | | D7237-10 | OIA-1677-09 ⁴⁴ |
| | Manual micro-diffusion and colorimetry | | | D4282-02 | |
| 25. Fluoride—Total, mg/L | Manual distillation, ⁶ followed by any of the following: | | 4500-F ⁻ B-19 97 | | |
| | Electrode, manual (ISE) | | 4500-F ⁻ C-19 97 | D1179-04 (B) | |
| | Electrode, automated (ISE) | | | | I-4327-85 ² |
| | Colorimetric, (SPADNS) | | 4500-F ⁻ D-19 97 | D1179-04 (A) | |
| | Automated complexone | | 4500-F ⁻ E-19 97 | | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 ³ |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 ⁵⁴ |
| 26. Gold—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 231.2 (Issued 1978) ¹ | 3113 B-2004 | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---|--|--|---|-------------------|---|
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| 27. Hardness—Total, as CaCO ₃ , mg/L | Automated colorimetric | 130.1 (Issued 1971) ¹ | | | |
| | Titrimetric (EDTA) | | 2340 C–1997 | D1126–02(07) | 973.52B, ³ I–1338–85 ² |
| | Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33). | | 2340 B–1997 | | |
| 28. Hydrogen ion (pH), pH units | Electrometric measurement | | 4500–H ⁺ B–2000 | D1293–99 (A or B) | 973.41, ³ I–1586–85 ² |
| | Automated electrode | 150.2 (Dec. 1982) ¹ | | | See footnote, ²¹ I–2587–85 ² |
| 29. Iridium—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B–1999 | | |
| | Graphite furnace AA (GFAA) | 235.2 (Issued 1978) ¹ | | | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | | 3125 B–2009 | | |
| 30. Iron—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B–1999 or 3111 C–1999 | D1068–05 (A or B) | 974.27, ³ I–3381–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D1068–05 (C) | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Phenanthroline) | | 3500–Fe B–1997 | D1068–05 (D) | Note ²² |
| 31. Kjeldahl Nitrogen ⁵ —Total, (as N), mg/L | Manual digestion ²⁰ and distillation or gas diffusion, followed by any of the following: | | 4500–N _{org} -B–1997 or C–1997 and 4500–NH ₃ B–1997 | D3590–02(06) (A) | I–4515–91 ⁴⁵ |
| | Titration | | 4500–NH ₃ C–1997 | | 973.48 ³ |
| | Electrode | | 4500–NH ₃ D–1997 or E–1997 | D1426–08 (B) | |
| | Semi-automated phenate | 350.1 Rev 2.0 1993 | 4500–NH ₃ G–1997, 4500–NH ₃ H–1997 | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|--|--|---------------------------------|-------------------|---|
| | Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods | | 4500-NH ₃ F-1 997 | | Note ⁶⁰ |
| Automated Methods for TKN that do not require manual distillation | | | | | |
| | Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation) | 351.1 (Rev. 1978) ¹ | | | I-4551-78. ⁸ |
| | Semi-automated block digester colorimetric (distillation not required) | 351.2, Rev. 2.0 (1993) | 4500-N _{org} -D-1997 | D3590-02(06) (B) | I-4515-91 ⁴⁵ |
| | Block digester, followed by Auto distillation and Titration | | | | Note ³⁹ |
| | Block Digester, followed by Flow injection gas diffusion (distillation not required) | | | | Note ⁴¹ |
| 32. Lead—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B-1999 or 3111 C-1999. | D3559-08 (A or B) | 974.27, ³ I-3399-85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D3559-08 (D) | I-4403-89 ⁵¹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma-atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 ⁵⁰ |
| | Inductively coupled plasma-mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14, ³ I-4471-97 ⁵⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190-08 | Note ³⁴ |
| | Voltametry ¹¹ | | | D3559-08 (C) | |
| | Colorimetric (Dithizone) | | 3500-Pb B-1997 | | |
| 33. Magnesium—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | D511-08 (B) | 974.27, ³ I-3447-85 ² |
| | Inductively coupled plasma-atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B-1999 | D1976-07 | I-4471-97 ⁵⁰ |
| | Inductively coupled plasma-mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 ³ |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| | Ion Chromatography | | | D6919-09 | |
| 34. Manganese—Total ⁴ , mg/L | Digestion ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B-1999 | D858-07 (A or B) | 974.27, ³ I-3454-85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | D858-07 (C) | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |

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List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---|---|--|--|-----------------------------|---|
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Persulfate) | | 3500–Mn B–1999 | | 920.203 ³ |
| | Colorimetric (Periodate) | | | | Note ²³ |
| 35. Mercury—Total, ⁴ mg/L | Cold vapor, Manual | 245.1, Rev. 3.0 (1994) | 3112 B–2009 | D3223–02(07) | 977.22, ³ I–3462–85 ² |
| | Cold vapor, Automated | 245.2 (Issued 1974) ¹ | | | |
| | Cold vapor atomic fluorescence spectrometry (CVAFS) | 245.7 Rev. 2.0 (2005) ¹⁷ | | | I–4464–01 ⁷¹ |
| | Purge and Trap CVAFS | 1631E ⁴³ | | | |
| 36. Molybdenum—Total, ⁴ mg/L | Digestion, ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D–1999 | | I–3490–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | | I–3492–96 ⁴⁷ |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| 37. Nickel—Total, ⁴ mg/L | Digestion ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B–1999 or 3111 C–1999 | D1886–08 (A or B) | I–3499–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D1886–08 (C) | I–4503–89 ⁵¹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14, ³ I–4020–05 ⁷⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | 38. Nitrate (as N), mg/L | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997) | 4110 B–2000 or C–2000 | D4327–03 |
| Capillary ion electrophoresis (CIE/UV) | | | 4140 B–1997 | D6508–00(05) | D6508, Rev. 2 ⁵⁴ |
| Ion Selective Electrode | | | 4500–NO ₃ –D–2000 | | |

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List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|---|---|---|--------------|--|
| | Nitrate–nitrite N minus Nitrite N (See parameters 39 and 40) | | | | Note ⁶² |
| 39. Nitrate + nitrite (as N), mg/L | Cadmium reduction, Manual | | 4500–NO ₃ ⁻ E–2000 | D3867–04 (B) | |
| | Cadmium reduction, Automated | 353.2, Rev. 2.0 (1993) | 4500–NO ₃ ⁻ F–2000 | D3867–04 (A) | I–2545–90 ⁵¹ |
| | Automated hydrazine | | 4500–NO ₃ ⁻ H–2000 | | |
| | Reduction/Colorimetric | | | | Note ⁶² |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997) | 4110 B–2000 or C–2000 | D4327–03 | 993.30 ³ |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B–1997 | D6508–00(05) | D6508, Rev. 2 ⁵⁴ |
| 40. Nitrite (as N), mg/L | Spectrophotometric: Manual | | 4500–NO ₂ ⁻ B–2000 | | Note ²⁵ |
| | Automated (Diazotization) | | | | I–4540–85 ² , Note ⁶² |
| | Automated (*bypass cadmium reduction) | 353.2, Rev. 2.0 (1993) | 4500–NO ₃ ⁻ F–2000 | D3867–04 (A) | I–4545–85 ² |
| | Manual (*bypass cadmium reduction) | | 4500–NO ₃ ⁻ E–2000 | D3867–04 (B) | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997) | 4110 B–2000 or C–2000 | D4327–03 | 993.30 ³ |
| | Capillary ion electrophoresis (CIE/UV) | | 4140 B–1997 | D6508–00(05) | D6508, Rev. 2 ⁵⁴ |
| 41. Oil and grease—Total recoverable, mg/L | Hexane extractable material (HEM): n–Hexane extraction and gravimetry | 1664 Rev. A; 1664 Rev. B ⁴² | 5520 B–2001 ³⁸ | | |
| | Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry | 1664 Rev. A; 1664 Rev. B ⁴² | 5520 B–2001 ³⁸ and 5520 F–2001 ³⁸ | | |
| 42. Organic carbon—Total (TOC), mg/L | Combustion | | 5310 B–2000 | D7573–09 | 973.47 ³ , p. 14 ²⁴ |
| | Heated persulfate or UV persulfate oxidation | | 5310 C–2000 5310 D–2000 | D4839–03 | 973.47 ³ , p. 14 ²⁴ |
| 43. Organic nitrogen (as N), mg/L | Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4) | | | | |
| 44. Ortho–phosphate (as P), mg/L | Colorimetry, Ascorbic acid, Automated | 365.1, Rev. 2.0 (1993) | 4500–P F–1999 or G–1999 | | 973.56 ³ , I–4601–85 ² |
| | Colorimetry, Ascorbic Acid, Manual single reagent | | 4500–P E–1999 | D515–88(A) | 973.55 ³ |
| | Colorimetry, Ascorbic Acid, Manual two reagent | 365.3 (Issued 1978) ¹ | | | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997) | 4110 B–2000 or C–2000 | D4327–03 | 993.30 ³ |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|---|----------------------------------|---|-------------------|---|
| | Capillary ion electrophoresis (CIE/ UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 ⁵⁴ |
| 45. Osmium—Total ⁴ , mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D-1999 | | |
| | Graphite furnace AA (GFAA) | 252.2 (Issued 1978) ¹ | | | |
| 46. Oxygen, dissolved, mg/L | Winkler (Azide modification) | | 4500-O B-2001, C-2001, D-2001, E-2001, F-2001 | D888-09 (A) | 973.45B ³ , I-1575-78 ⁸ |
| | Electrode | | 4500-O G-2001 | D888-09 (B) | I-1576-78 ⁸ |
| | Luminescence Based Sensor | | | D888-09 (C) | Note ⁶³ Note ⁶⁴ |
| 47. Palladium—Total, ⁴ mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 253.2 ¹ (Issued 1978) | | | |
| | Inductively coupled plasma-mass spectrometry (ICP/MS) | | 3125 B-2009 | | |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| 48. Phenols, mg/L | Manual distillation ²⁶ , followed by any of the following: | 420.1 ¹ (Rev. 1978) | 5530 B-2005 | D1783-01 | |
| | Colorimetric (4AAP) manual | 420.1 ¹ (Rev. 1978) | 5530 D-2005 ²⁷ | D1783-01 (A or B) | |
| | Colorimetric (4AAP), Automated | 420.4 Rev. 1.0 (1993) | | | |
| 49. Phosphorus (elemental), mg/L | Gas-liquid chromatography | | | | Note ²⁸ |
| 50. Phosphorus—Total, mg/L | Digestion ²⁰ , followed by any of the following: | | 4500-P B(5)-1999 | | 973.55 ³ |
| | Colorimetric, Manual ascorbic acid | 365.3 ¹ (Issued 1978) | 4500-P E-1999 | D515-88 (A) | |
| | Colorimetric, Automated ascorbic acid reduction | 365.1 Rev. 2.0 (1993) | 4500-P F-1999, G-1999, H-1999 | | 973.56 ³ , I-4600-85 ² |
| | Colorimetric, Semi-automated block digester (TKP digestion) | 365.4 ¹ (Issued 1974) | | D515-88 (B) | I-4610-91 ⁴⁸ |
| 51. Platinum—Total, ⁴ mg/L | Digestion ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 255.2 (Issued 1978) ¹ | | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|---|----------------------------------|------------------|--------------------|---|
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | | 3125 B–2009 | | |
| | Direct current plasma (DCP) | | | Note ³⁴ | |
| 52. Potassium—Total, ⁴ mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B–1999 | | 973.53 ³ , I–3630–85 ² |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994) | 3120 B–1999 | | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ |
| | Flame photometric | | 3500–K B–1997 | | |
| | Electrode | | 3500–K C–1997 | | |
| | Ion Chromatography | | | D6919–09 | |
| 53. Residue—Total, mg/L | Gravimetric, 103–105°C | | 2540 B–1997 | | I–3750–85 ² |
| 54. Residue—filterable (TDS), mg/L | Gravimetric, 180°C | | 2540 C–1997 | D5907–03 | I–1750–85 ² |
| 55. Residue—non-filterable (TSS), mg/L | Gravimetric, 103–105°C post washing of residue | | 2540 D–1997 | D5907–03 | I–3765–85 ² |
| 56. Residue—settleable, mg/L | Volumetric, (Imhoff cone), or gravimetric | | 2540 F–1997 | | |
| 57. Residue—Volatile, mg/L | Gravimetric, 550°C | 160.4 (Issued 1971) ¹ | 2540–E–1997 | | I–3753–85 ² |
| 58. Rhodium—Total, ⁴ mg/L | Digestion ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA), or | | 3111 B–1999 | | |
| | Graphite furnace AA (GFAA) | 265.2 (Issued 1978) ¹ | | | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | | 3125 B–2009 | | |
| 59. Ruthenium—Total, ⁴ mg/L | Digestion ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA), or | | 3111 B–1999 | | |
| | Graphite furnace AA (GFAA) | 267.2 ¹ | | | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8 | 3125 B–2009 | | |
| 60. Selenium—Total, ⁴ mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D3859–08 (B) | I–4668–98 ⁴⁹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---|---|--|---|------------------|---|
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ , I–4020–05 ⁷⁰ |
| | AA gaseous hydride | | 3114 B–2009, or 3111 C–2009 | D3859–08 (A) | I–3667–85 ² |
| 61. Silica—Dis-solved, ³⁷ mg/L | 0.45–micron filtration followed by any of the following: | | | | |
| | Colorimetric, Manual | | 4500–SiO ₂ C–1997 | D859–05 | I–1700–85 ² |
| | Colorimetric, Automated (Molybdo-silicate) | | 4500–SiO ₂ E–1997 or F–1997 | | I–2700–85 ² |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ |
| 62. Silver—Total, ⁴ , ³¹ mg/L | Digestion ⁴ , ²⁹ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B–1999 or 3111 C–1999 | | 974.27 ³ , p. 37 ⁹ , I–3720–85 ² |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | | I–4724–89 ⁵¹ |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ , I–4471–97 ⁵⁰ |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| 63. Sodium—Total, ⁴ m-g/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B–1999 | | 973.54 ³ , I–3735–85 ² |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| | Flame photometric | | 3500–Na B–1997 | | |
| | Ion Chromatography | | | D6919–09 | |
| 64. Specific con-ductance, micromhos/cm at 25 °C | Wheatstone bridge | 120.1 ¹ (Rev. 1982) | 2510 B–1997 | D1125–95(99) (A) | 973.40 ³ , I–2781–85 ² |
| 65. Sulfate (as SO ₄), mg/L | Colorimetric, Automated | 375.2, Rev. 2.0 (1993) | 4500–SO ₄ ²⁻ F–1997 or G–1997 | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|--|--|--|---|------------------|--|
| | Gravimetric | | 4500-SO ₄ ²⁻ -C -1997 or D-1997 | | 925.54 ³ |
| | Turbidimetric | | 4500-SO ₄ ²⁻ -E -1997 | D516-07 | |
| | Ion Chromatography | 300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997) | 4110 B-2000 or C-2000 | D4327-03 | 993.30 ³ , I-4020-05 ⁷⁰ |
| | Capillary ion electrophoresis (CIE/ UV) | | 4140 B-1997 | D6508-00(05) | D6508, Rev. 2 ⁵⁴ |
| 66. Sulfide (as S), mg/L | Sample Pretreatment | | 4500-S ²⁻ -B, C-2000 | | |
| | Titrimetric (iodine) | | 4500-S ²⁻ -F-20 00 | | I-3840-85 ² |
| | Colorimetric (methylene blue) | | 4500-S ²⁻ -D-2 000 | | |
| | Ion Selective Electrode | | 4500-S ²⁻ -G-2 000 | D4658-08 | |
| 67. Sulfite (as SO ₃), mg/L | Titrimetric (iodine-iodate) | | 4500-SO ₃ ²⁻ -B -2000 | | |
| 68. Surfactants, mg/L | Colorimetric (methylene blue) | | 5540 C-2000 | D2330-02 | |
| 69. Temperature, °C | Thermometric | | 2550 B-2000 | | Note ³² |
| 70. Thallium-Tot- al, ⁴ mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | |
| | Graphite furnace AA (GFAA) | 279.2 ¹ (Issued 1978) | 3113 B-2004 | | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma-atomic emission spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994); 200.5 Rev. 4.2 (2003) ⁶⁸ | 3120 B-1999 | D1976-07 | |
| | Inductively coupled plasma-mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 ³ , I-4471-97 ⁵⁰ |
| 71. Tin-Total, ⁴ mg/ L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 B-1999 | | I-3850-78 ⁸ |
| | Graphite furnace AA (GFAA) | | 3113 B-2004 | | |
| | Stabilized temperature GFAA (STGFAA) | 200.9, Rev. 2.2 (1994) | | | |
| | Inductively coupled plasma-atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | | | |
| | Inductively coupled plasma-mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B-2009 | D5673-05 | 993.14 ³ |
| 72. Titanium-Tot- al, ⁴ mg/L | Digestion ⁴ followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D-1999 | | |

Table B
List of Approved Inorganic Test Procedures For Wastewater

| Parameter, Units | Analytical Technology ⁵⁸ | EPA ⁵² | Standard methods | ASTM | USGS AOAC Other |
|---------------------------------------|---|--|----------------------------|-----------------------|--|
| | Graphite furnace AA (GFAA) | 283.2 ¹ (Issued 1978) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.7, Rev. 4.4 (1994) | | | |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ |
| | Direct current plasma (DCP) | | | | Note ³⁴ |
| 73. Turbidity, NTU ⁵³ | Nephelometric | 180.1, Rev. 2.0 (1993) | 2130 B–2001 | D1889–00 | I–3860–85 ² Note ⁶⁵ Note ⁶⁶ Note ⁶⁷ |
| 74. Vanadium–Total, ⁴ mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) | | 3111 D–1999 | | |
| | Graphite furnace AA (GFAA) | | 3113 B–2004 | D3373–03(07) | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ , I–4020–05 ⁷⁰ |
| | Direct current plasma (DCP) | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Gallic Acid) | | 3500–V B–1997 | | |
| 75. Zinc–Total ⁴ , mg/L | Digestion ⁴ , followed by any of the following: | | | | |
| | AA direct aspiration (FLAA) ³⁶ | | 3111 B–1999 or 3111 C–1999 | D1691–02(07) (A or B) | 974.27 ³ , p. 37 ⁹ , I–3900–85 ² |
| | Graphite furnace AA (GFAA) | 289.2 ¹ (Issued 1978) | | | |
| | Inductively coupled plasma–atomic emission spectrometry (ICP/AES) ³⁶ | 200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994) | 3120 B–1999 | D1976–07 | I–4471–97 ⁵⁰ |
| | Inductively coupled plasma–mass spectrometry (ICP/MS) | 200.8, Rev. 5.4 (1994) | 3125 B–2009 | D5673–05 | 993.14 ³ I–4020–05 ⁷⁰ |
| | Direct current plasma (DCP) ³⁶ | | | D4190–08 | Note ³⁴ |
| | Colorimetric (Zincon) | | 3500 Zn B–1997 | | Note ³³ |
| 76. Acid Mine Drainage | | 1627 ⁶⁹ | | | |

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

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

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

4 For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic–metal complexes (to convert the analyte to a detectable form for colorimetric analysis).

For non–platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness.

For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R-94/111, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions.

For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP-AES, and ICP-MS) use EPA Method 200.2 or an approved alternate procedure (e.g., CEM microwave digestion, which may be used with certain analytes as indicated in Table IB); the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.

- 5 Copper sulfate or other catalysts that have been found suitable may be used in place of mercuric sulfate.
- 6 Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step. (A total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as < 20% RPD for all tested matrices). Alternatively the two populations of spike recovery percentages may be compared using a recognized statistical test.
- 7 Industrial Method Number 379-75 WE Ammonia, Automated Electrode Method, Technicon Auto Analyzer II. February 19, 1976. Bran & Luebbe Analyzing Technologies Inc.
- 8 The approved method is that cited in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. USGS.
- 9 American National Standard on Photographic Processing Effluents. April 2, 1975. American National Standards Institute (ANSI), 25 West 43rd St., New York, NY 10036.
- 10 In-Situ Method 1003-8-2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. 2009. In-Situ Incorporated.
- 11 The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
- 12 Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD₅ is required can the permittee report data using a nitrification inhibitor.
- 13 OIC Chemical Oxygen Demand Method. 1978. Oceanography International Corporation. 512 West Loop, P.O. Box 2980, College Station, TX 77840.
- 14 Method 8000, Chemical Oxygen Demand, Hach Handbook of Water Analysis, 1979. Hach Company. P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- 15 The back titration method will be used to resolve controversy.
- 16 Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70. Thermo Scientific, 81 Wyman Street, Waltham, MA 02454. 1977. Orion Research Incorporated. The calibration for the Orion residual chlorine method must be derived using at least three standard solutions, prepared from a 0.00281 N potassium iodate solution.
- 17 Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-05-001. Revision 2.0, February 2005. US EPA., available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304.
- 18 National Council of the Paper Industry for Air and Stream Improvement (NCASI) Technical Bulletin 253, December 1971.
- 19 Method 8506, Biocinchoninate Method for Copper, Hach Handbook of Water Analysis. 1979. Hach Company. P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- 20 When using a method with block digestion, this treatment is not required.
- 21 Industrial Method Number 378-75WA, Hydrogen ion (pH) Automated Electrode Method, Bran & Luebbe (Technicon) Autoanalyzer II. October 1976. Bran & Luebbe Analyzing Technologies. Elmsford, NY 10523.
- 22 Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water. 1980. Hach Company P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- 23 Method 8034, Periodate Oxidation Method for Manganese, Hach Handbook of Wastewater Analysis. 1979. Hach Company Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- 24 Methods for Analysis of Organic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14. 1987. USGS. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- 25 Method 8507, Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater. 1979. Hach Company P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- 26 Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.
- 27 The colorimetric reaction must be conducted at a pH of 10.0 ± 0.2.
- 28 Addison, R.F., and R.G. Ackman. 1970. Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography, *Journal of Chromatography*, 47(3):421-426. Available in most public libraries. Back volumes of the Journal of Chromatography are available from: Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

- ²⁹ Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na₂S₂O₃ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
- ³⁰ The use of EDTA decreases method sensitivity. Analysts may omit EDTA or replace with another suitable complexing reagent provided that all method specified quality control acceptance criteria are met.
- ³¹ For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH₄OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I₂ to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH>7 with NH₄OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100–mL volumetric flask and dilute to volume with water.
- ³² “Water Temperature—Influential Factors, Field Measurement and Data Presentation,” Techniques of Water—Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1. 1975. USGS. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- ³³ Method 8009, Zincon Method for Zinc, Hach Handbook of Water Analysis, 1979. Hach Company. Loveland, CO 80537. Available on–line at <http://www.hach.com>.
- ³⁴ Method AES0029, Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes. 1986—Revised 1991. Thermo Jarrell Ash Corporation. Available from: Thermo Scientific, 81 Wyman Street, Waltham, MA 02454.
- ³⁵ In–Situ Method 1004–8–2009, Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe. 2009. In–Situ Incorporated.
- ³⁶ Microwave–assisted digestion may be employed for this metal, when analyzed by this methodology. Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals. April 16, 1992. CEM Corporation, P.O. Box 200, Matthews, NC 28106–0200.
- ³⁷ When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.
- ³⁸ Only use n–hexane (n–Hexane—85% minimum purity, 99.0% min. saturated C6 isomers, residue less than 1 mg/L) extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Methods 1664 Rev. A and 1664 Rev. B). Use of other extraction solvents is prohibited.³⁹ Method PAI–DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric Detection. Revised December 22, 1994. OI Analytical/ALP– KEM, P.O. Box 9010, College Station, TX 77842.
- ⁴⁰ Method PAI–DK02, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Colorimetric Detection. Revised December 22, 1994. OI Analytical.
- ⁴¹ Method PAI–DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion. Revised December 22, 1994. OI Analytical/ALP–KEM, P.O. Box 9010, College Station, TX 77842.
- ⁴² Method 1664 Rev. B is the revised version of EPA Method 1664 Rev. A. U.S. EPA. February 1999, Revision A. Method 1664, n–Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n–Hexane Extractable Material (SGT–HEM; Non–polar Material) by Extraction and Gravimetry. EPA–821–R–98–002. U.S. EPA. February 2010, Revision B. Method 1664, n–Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n–Hexane Extractable Material (SGT–HEM; Non–polar Material) by Extraction and Gravimetry. EPA–821–R–10–001. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.
- ⁴³ Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, EPA–821–R–02–019. Revision E. August 2002, U.S. EPA. The application of clean techniques described in EPA’s Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels*, EPA–821–R–96–011, are recommended to preclude contamination at low–level, trace metal determinations. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.
- ⁴⁴ Method OIA–1677–09, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA). 2010. OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- ⁴⁵ Open File Report 00–170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. USGS.
- ⁴⁶ Open File Report 93–449, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1993. USGS.
- ⁴⁷ Open File Report 97–198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry. 1997. USGS.
- ⁴⁸ Open File Report 92–146, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis. 1992. USGS.
- ⁴⁹ Open File Report 98–639, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace–Atomic Absorption Spectrometry. 1999. USGS.
- ⁵⁰ Open File Report 98–165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole–water Digests Using Inductively Coupled Plasma–Optical Emission Spectrometry and Inductively Coupled Plasma–Mass Spectrometry. 1998. USGS.
- ⁵¹ Open File Report 93–125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.
- ⁵² Unless otherwise indicated, all EPA methods, excluding EPA Method 300.1–1, are published in U.S. EPA. May 1994. Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA/600/R–94/111; or U.S. EPA. August 1993. Methods for the Determination

of Inorganic Substances in Environmental Samples, EPA/600/R-93/100. EPA Method 300.1 is US EPA. Revision 1.0, 1997, including errata cover sheet April 27, 1999. Determination of Inorganic Ions in Drinking Water by Ion Chromatography.

- 53 Styrene divinyl benzene beads (e.g., AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StabCal™ or equivalent) are acceptable substitutes for formazin.
- 54 Method D6508, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. December 2000. Waters Corp., 34 Maple St., Milford, MA, 01757, Telephone: 508/482-2131, Fax: 508/482-3625.
- 55 Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate, EPA 821-B-01-009, Revision 1.2, August 2001. US EPA. National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001-108275]. The toll free telephone number is: 800-553-6847.
Note: A 450-W UV lamp may be used in this method instead of the 550-W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.
- 56 QuikChem Method 10-204-00-1-X, Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis. Revision 2.2, March 2005. Lachat Instruments. Available from Hach Company, P.O. Box 389, Loveland, CO 80537.
- 57 When using sulfide removal test procedures described in EPA Method 335.4-1, reconstitute particulate that is filtered with the sample prior to distillation.
- 58 Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation “followed by” analysis with a method, approved digestion and/or distillation are required prior to analysis.
- 59 Samples analyzed for available cyanide using OI Analytical method OIA-1677-09 or ASTM method D6888-09 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample filtration to no more than 30 minutes to preclude settling of materials in samples.
- 60 Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction (“phenol-hypochlorite reaction”) colorimetric ammonium determination methods. For example when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, Patton, C.J. and S.R. Crouch. March 1977. Anal. Chem. 49:464-469. These reaction parameters increase to pH > 12.6 and 665 nm when salicylate is used as the color reagent—see, Krom, M.D. April 1980. The Analyst 105:305-316.
- 61 If atomic absorption or ICP instrumentation is not available, the aluminon colorimetric method detailed in the 19th Edition of *Standard Methods* may be used. This method has poorer precision and bias than the methods of choice.
- 62 Easy (1-Reagent) Nitrate Method, Revision November 12, 2011. Craig Chinchilla.
- 63 Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and cBOD₅. Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).
- 64 In-Situ Method 1002-8-2009, Dissolved Oxygen (DO) Measurement by Optical Probe. 2009. In-Situ Incorporated.
- 65 Mitchell Method M5331, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.
- 66 Mitchell Method M5271, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.
- 67 Orion Method AQ4500, Determination of Turbidity by Nephelometry. Revision 5, March 12, 2009. Thermo Scientific.
- 68 EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA/600/R-06/115. Revision 4.2, October 2003. US EPA.
- 69 Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality, EPA-821-R-09-002. December 2011. US EPA.
- 70 Techniques and Methods Book 5-B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry, Chapter 1, Section B, Methods of the National Water Quality Laboratory, Book 5, Laboratory Analysis, 2006. USGS.
- 71 Water-Resources Investigations Report 01-4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water With Cold Vapor-Atomic Fluorescence Spectrometry, 2001. USGS.
- 72 Quality control requirements for low level mercury are found in s. NR 106.145 (9) and (10), Wis. Adm. Code. Low-level mercury methods are performance based so some method modifications are allowable, provided quality control requirements are met. If an atomic absorption detector is substituted for atomic fluorescence detector, the appropriate method citation is 245.1 (manual) or 245.2 (automated). If method 1631E is modified to eliminate the purge and trap step, the appropriate method citation is 245.7.

Table C
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| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|------------------------|--------------------------|--------------------|---------------------|------|------------------------------|
| 1. Acenaphthene | GC | 610 | | | |
| | GC/MS | 625,1625B | 6410 B-2000 | | Note ⁹ , p. 27 |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|---------------------------------|-----------------------|--------------------------|------------------|---------------|-----------------------------|
| 2. Acenaphthylene | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| | GC | 610 | | | |
| | GC/MS | 625,1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 3. Acrolein | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| | GC | 603 | | | |
| 4. Acrylonitrile | GC/MS | 624 ⁴ ,1624B | | | |
| | GC | 603 | | | |
| 5. Anthracene | GC/MS | 624 ⁴ , 1624B | | | |
| | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 6. Benzene | HPLC | 610 | 6440B-2000 | D4657-92 (98) | |
| | GC | 602 | 6200 C-1997 | | |
| 7. Benzidine | GC/MS | 624, 1624B | 6200 B-1997 | | |
| | Spectro-photometric | | | | Note ³ , p.1 |
| 8. Benzo(a)anthracene | GC/MS | 625 ⁵ , 1625B | 6410 B-2000 | | |
| | HPLC | 605 | | | |
| | GC | 610 | | | |
| 9. Benzo(a)pyrene | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| | GC | 610 | | | |
| 10. Benzo(b)fluoranthene | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| | GC | 610 | | | |
| 11. Benzo(g,h,i)perylene | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| | GC | 610 | | | |
| 12. Benzo(k)fluoranthene | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| | GC | 610 | | | |
| 13. Benzyl chloride | GC/MS | | | | Note ³ , p. 130 |
| | GC | | | | Note ⁶ , p. S102 |
| 14. Butyl benzyl phthalate | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | GC | 606 | | | |
| 15. bis(2-Chloroethoxy) methane | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | GC | 611 | | | |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|---------------------------------|-----------------------|--------------------|------------------|------|----------------------------|
| 16. bis(2-Chloroethyl) ether | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 17. bis(2-Ethylhexyl) phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 18. Bromodichloromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 19. Bromoform | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 20. Bromomethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 21. 4-Bromophenyl phenyl ether | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 22. Carbon tetrachloride | GC | 601 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 23. 4-Chloro-3-methyl phenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 24. Chlorobenzene | GC | 601, 602 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 25. Chloroethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 26. 2-Chloroethylvinyl ether | GC | 601 | | | |
| | GC/MS | 624, 1624B | | | |
| 27. Chloroform | GC | 601 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 28. Chloromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 29. 2-Chloronaphthalene | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 30. 2-Chlorophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 31. 4-Chlorophenyl phenyl ether | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|--------------------------------------|-----------------------|--------------------|------------------|---------------|---------------------------|
| 32. Chrysene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 33. Dibenzo(a,h)anthracene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 34. Dibromochloromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 35. 1,2-Dichlorobenzene | GC | 601, 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1625B | 6200 B-1997 | | Note ⁹ , p. 27 |
| 36. 1,3-Dichlorobenzene | GC | 601, 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1625B | 6200 B-1997 | | Note ⁹ , p. 27 |
| 37. 1,4-Dichlorobenzene | GC | 601, 602 | 6200 C-1997. | | |
| | GC/MS | 624, 1625B | 6200 B-1997 | | Note ⁹ , p. 27 |
| 38. 3,3'-Dichlorobenzidine | GC/MS | 625, 1625B | 6410 B-2000 | | |
| | HPLC | 605 | | | |
| 39. Dichlorodifluoromethane | GC | 601 | | | |
| | GC/MS | | 6200 C-1997 | | |
| 40. 1,1-Dichloroethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 41. 1,2-Dichloroethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 42. 1,1-Dichloroethene | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 43. <i>trans</i> -1,2-Dichloroethene | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 44. 2,4-Dichlorophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 45. 1,2-Dichloropropane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 46. <i>cis</i> -1,3-Dichloropropene | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|-------------------------------|-----------------------|--------------------|------------------|---------------|-----------------------------|
| 47. trans-1,3-Dichloropropene | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 48. Diethyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 49. 2,4-Dimethylphenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 50. Dimethyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 51. Di-n-butyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 52. Di-n-octyl phthalate | GC | 606 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 53. 2, 3-Dinitrophenol | GC | 604 | 6420 B-2000 | | Note ⁹ , p. 27 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | |
| 54. 2, 4-Dinitrophenol | GC | 604 | 6420 B-2000 | | Note ⁹ , p. 27 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | |
| 55. 2, 6-Dinitrophenol | GC | 604 | 6420 B-2000 | | Note ⁹ , p. 27 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | |
| 56. 2,3-Dinitrotoluene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 57. 2,4-Dinitrotoluene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 58. 2,6-Dinitrotoluene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 59. Epichlorohydrin | GC | | | | Note ³ , p. 130 |
| | GC/MS | | | | Note ⁶ , p. S102 |
| 60. Ethylbenzene | GC | 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 61. Fluoranthene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|--|-----------------------|--------------------------|------------------|---------------|----------------------------|
| 62. Fluorene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 63. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran | HRGC/MS | 1613B | | | |
| 64. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran | HRGC/MS | 1613B | | | |
| 65. 1,2,3,4,6,7,8-Heptachloro-dibenzo-p-dioxin | HRGC/MS | 1613B | | | |
| 66. Hexachlorobenzene | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 67. Hexachlorobutadiene | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 68. Hexachlorocyclopentadiene | GC | 612 | | | |
| | GC/MS | 625 ⁵ , 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 69. 1,2,3,4,7,8-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 70. 1,2,3,6,7,8-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 71. 1,2,3,7,8,9-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 72. 2,3,4,6,7,8-Hexachloro-dibenzofuran | HR GC/MS | 1613B | | | |
| 73. 1,2,3,4,7,8-Hexachloro-dibenzo-p-dioxin | HR GC/MS | 1613B | | | |
| 74. 1,2,3,6,7,8-Hexachloro-dibenzo-p-dioxin | HR GC/MS | 1613B | | | |
| 75. 1,2,3,7,8,9-Hexachloro-dibenzo-p-dioxin | HR GC/MS | 1613B | | | |
| 76. Hexachloroethane | GC | 612 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 77. Indeno(1,2,3-c,d) pyrene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 78. Isophorone | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 79. Methylene chloride | GC | 601 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 80. 2-Methyl-4,6-dinitrophenol | GC | 604 | 6420 B-2000 | | |

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| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|--|-----------------------|--------------------------|------------------|---------------|---|
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 81. Naphthalene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000. | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000. | | |
| 82. Nitrobenzene | GC | 609 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | | | D4657-92 (98) | |
| 83. 2-Nitrophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 84. 4-Nitrophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 85. N-Nitrosodimethylamine | GC | 607 | | | |
| | GC/MS | 625 ⁵ , 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 86. N-Nitrosodi-n-propylamine | GC | 607 | | | |
| | GC/MS | 625 ⁵ , 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 87. N-Nitrosodiphenylamine | GC | 607 | | | |
| | GC/MS | 625 ⁵ , 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 88. Octachlorodibenzofuran | HR GC/MS | 1613B ¹⁰ | | | |
| 89. Octachlorodibenzo-p-dioxin | HR GC/MS | 1613B ¹⁰ | | | |
| 90. 2,2'-Oxybis(2-chloro-propane) [prev.: bis(2-Chloroisopropyl) ether] | GC | 611 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 91. PCB-1016, (Aroclor or congeners) ^{12,13} | GC | 608 | | | Note ³ , p. 43; Note ⁸ |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HR GC/MS | 1668A ¹⁴ | | | |
| 92. PCB-1221, (Aroclor or congeners) ^{12,13} | GC | 608 | | | Note ³ , p. 43; Note ⁸ |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A ¹⁴ | | | |
| 93. PCB-1232, (Aroclor or congeners) ^{12,13} | GC | 608 | | | Note ³ , p. 43; Note ⁸ |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/ MS | 1668A ¹⁴ | | | |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|---|-----------------------|--------------------------|------------------|---------------|---|
| 94. PCB-1242, (Aroclor or congeners) ^{12,13} | GC | 608 | | | Note ³ , p. 43; Note ⁸ |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/MS | 1668A ¹⁴ | | | |
| 95. PCB-1248, (Aroclor or congeners) ^{12,13} | GC | 608 | | | |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/MS | 1668A ¹⁴ | | | |
| 96. PCB-1254, (Aroclor or congeners) ^{12,13} | GC | 608 | | | Note ³ , p. 43; Note ⁸ |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/MS | 1668A ¹⁴ | | | |
| 97. PCB-1260, (Aroclor or congeners) ^{12,13} | GC | 608 | | | Note ³ , p. 43; Note ⁸ |
| | GC/MS | 625 | 6410 B-2000 | | |
| | HRGC/MS | 1668A ¹⁴ | | | |
| 98. 1,2,3,7,8-Pentachloro-dibenzofuran | GC/MS | 1613B | | | |
| 99. 2,3,4,7,8-Pentachloro-dibenzofuran | GC/MS | 1613B | | | |
| 100. 1,2,3,7,8,-Pentachloro-dibenzo-p-dioxin | GC/MS | 1613B | | | |
| 101. Pentachlorophenol | GC | 604 | 6420 B-2000 | | Note ³ , p. 140 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 102. Phenanthrene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 103. Phenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 104. Pyrene | GC | 610 | | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| | HPLC | 610 | 6440 B-2000 | D4657-92 (98) | |
| 105. 2,3,7,8-Tetrachloro-dibenzofuran | HR GC/MS | 1613B ¹⁰ | | | |
| 106. 2,3,7,8-Tetrachloro-dibenzo-p-dioxin | GC/MS | 613, 625 ^{5a} , | | | |
| 107. 1,1,2,2-Tetrachloroethane | GC | 601 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 108 Tetrachlorocatechol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 109. Tetrachloroethene | GC | 601 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |

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

| Parameter ¹ | Analytical Technology | EPA ^{2,7} | Standard methods | ASTM | Other |
|---|--------------------------------------|--------------------|------------------|----------|----------------------------|
| 110. Tetrachloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 111. 2,3,4,6- Tetrachlorophenol | GC | | 6420 B-2000 | | |
| | GC/MS | | 6410 B-2000 | | |
| 112. Toluene | GC | 602 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 113. 1,2,4-Trichlorobenzene | GC | 612 | | | Note ³ , p. 130 |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 114. 3,4,5-Trichlorocatechol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 115. 3,4,6-Trichlorocatechol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 116. 1,1,1-Trichloroethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 117. 1,1,2-Trichloroethane | GC | 601 | 6200 C-1997 | | Note ³ , p. 130 |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 118. Trichloroethene | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 119. Trichlorofluoromethane | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624 | 6200 B-1997 | | |
| 120. 3,4,5-Trichloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 121. 3,4,6-Trichloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 122. 4,5,6-Trichloroguaicol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 123. 2,4,5-Trichlorophenol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 124. 2,4,6-Trichlorophenol | GC | 604 | 6420 B-2000 | | |
| | GC/MS | 625, 1625B | 6410 B-2000 | | Note ⁹ , p. 27 |
| 125. Trichlorosyringol | GC | | 6420 B-2000 | | |
| | GC/MS | 1653 ¹¹ | 6410 B-2000 | | |
| 126. Vinyl chloride | GC | 601 | 6200 C-1997 | | |
| | GC/MS | 624, 1624B | 6200 B-1997 | | |
| 127. Nonylphenol | GC/MS | | | D7065-06 | |
| 128. Bisphenol A (BPA) | GC/MS | | | D7065-06 | |
| 129. p-tert-Octylphenol (OP) | GC/MS | | | D7065-06 | |
| 130. Nonylphenol Monoethoxylate (NP1EO) | GC/MS | | | D7065-06 | |
| 131. Nonylphenol Diethoxylate (NP2EO) | GC/MS | | | D7065-06 | |
| 132. Adsorbable Organic Halides (AOX) | Adsorption and Coulometric Titration | 1650 ¹¹ | | | |
| 133. Chlorinated Phenolics | In Situ Acetylation and GC/MS | 1653 ¹¹ | | | |

- ¹ All parameters are expressed in micrograms per liter ($\mu\text{g/L}$) except for Method 1613B, in which the parameters are expressed in picograms per liter (pg/L).
- ² The full text of Methods 601-613, 624, 625, 1613B, 1624B, and 1625B are provided at Appendix A, Test Procedures for Analysis of Organic Pollutants, of 40 CFR Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at 40 CFR Part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit.
- ³ Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA.
- ⁴ Method 624 may be used for quantitative determination of acrolein and acrylonitrile, provided that the laboratory has documentation to substantiate the ability to detect and quantify these analytes at levels necessary to comply with any associated regulations. In addition, the use of sample introduction techniques other than simple purge-and-trap may be required. QC acceptance criteria from Method 603 should be used when analyzing samples for acrolein and acrylonitrile in the absence of such criteria in Method 624.
- ⁵ Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, N-nitrosodi-n-propylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.
- ^{5a} Method 625, screening only.
- ⁶ Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the 15th Edition of *Standard Methods for the Examination of Water and Wastewater*. 1981. American Public Health Association (APHA).
- ⁷ Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601-603, 624, 625, 1624B, and 1625B in accordance with procedures each in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624B and 1625B) of all samples to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.
- ⁸ Organochlorine Pesticides and PCBs in Wastewater Using EmporeTMDisk. Revised October 28, 1994. 3M Corporation.
- ⁹ Method O-3116-87 is in Open File Report 93-125, Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.
- ¹⁰ Analysts may use Fluid Management Systems, Inc. Power-Prep system in place of manual cleanup provided the analyst meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities. Method 1613, Revision B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. Revision B, 1994. U.S. EPA. The full text of this method is provided in Appendix A to 40 CFR Part 136 and at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.
- ¹¹ Method 1650, Adsorbable Organic Halides by Adsorption and Coulometric Titration. Revision C, 1997. U.S. EPA. Method 1653, Chlorinated Phenolics in Wastewater by In Situ Acetylation and GCMS. Revision A, 1997. U.S. EPA. The full text for both of these methods is provided at Appendix A, "Methods 1650 and 1653", in Part 430, The Pulp, Paper, and Paperboard Point Source Category. Also available on-line at <http://www.gpo.gov/>.
- ¹² EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non-detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration reported. It is recognized a number of congeners will co-elute with others, so there will not be 209 results to sum.
- ¹³ If congener specific analysis is performed, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular sample. If Aroclor analysis is performed, clean up steps of the extract shall be performed as necessary to remove interference. If congener specific analysis is done, clean up steps of the extract shall be performed as necessary to remove interference. If desired limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:
Florisil, Gel Permeation, Silica Gel, Alumina, Sulfur Clean Up, Sulfuric Acid Clean Up.
- ¹⁴ "Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS", EPA-821-R-00-002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Table D
List of Approved Test Procedures for Pesticides ¹

| Parameter | Analytical Technology | EPA ^{2,7,10} | Standard Methods | ASTM | Other |
|---------------------|-----------------------|-----------------------|----------------------------|----------------------------|--|
| 1. Aldrin | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96 (02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 2. Ametryn | GC | 507, 619 | | | Note ³ , p. 83; Note ⁹ , O-3106-93; Note ⁶ , p. S68 |
| | GC/MS | 525.2 | | | Note ¹⁴ , O-1121-91 |
| 3. Aminocarb | TLC | | | | Note ³ , p. 94; Note ⁶ , p. S60 |
| | HPLC | 632 | | | |
| 4. Atraton | GC | 619 | | | Note ³ , p. 83; Note ⁶ , p. S68 |
| 5. Atrazine | GC | 507, 619 | | | Note ³ , p. 83; Note ⁶ , p. S68; Note ⁹ , O-3106-93 |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| | GC/MS | 525.1, 525.2 | | | Note ¹¹ , O-1126-95 |
| 6. Azinphos methyl | GC | 614, 622, 1657 | | | Note ³ , p. 25; Note ⁶ , p. S51 |
| | GC-MS | | | | Note ¹¹ , O-1126-95 |
| 7. Barban | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| 8. α-BHC | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁸ , 3M0222 |
| | GC/MS | 625 ⁵ | 6410 B-2000 | | Note ¹¹ , O-1126-95 |
| 9. β-BHC | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 10. δ-BHC | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 11. γ-BHC (Lindane) | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 ⁵ | 6410 B-2000 | | Note ¹¹ , O-1126-95 |
| 12. Captan | GC | 617 | 6630 B-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7 |
| 13. Carbaryl | TLC | | | | Note ³ , p. 94, Note ⁶ , p. S60 |
| | HPLC | 531.1, 632 | | | |
| | HPLC/MS | 553 | | | Note ¹² , O-2060-01 |
| | GC/MS | | | | Note ¹¹ , O-1126-95 |
| 14. Carbophenothion | GC | 617 | 6630 B-2000 | | Note ⁴ , page 27; Note ⁶ , p. S73 |
| 15. Chlordane | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 16. Chloropropham | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |

Table D
List of Approved Test Procedures for Pesticides ¹

| Parameter | Analytical Technology | EPA ^{2,7,10} | Standard Methods | ASTM | Other |
|------------------------|-----------------------|------------------------|----------------------------|---------------------------|--|
| | HPLC | 632 | | | |
| 17. 2,4-D | GC | 615 | 6640 B-2001 | | Note ³ , p. 115; Note ⁴ , O-3105-83 |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| 18. 4,4'-DDD | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3105-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000. | | |
| 19. 4,4'-DDE | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | Note ¹¹ , O-1126-95 |
| 20. 4,4'-DDT | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 21. Demeton-O | GC | 614, 622 | | | Note ³ , p. 25; Note ⁶ , p. S51 |
| 22. Demeton-S | GC | 614, 622 | | | Note ³ , p. 25; Note ⁶ , p. S51 |
| 23. Diazinon | GC | 507, 614, 622, 1657 | | | Note ³ , p. 25; Note ⁴ , O-3104-83; Note ⁶ , p. S51 |
| | GC/MS | 525.2 | | | Note ¹¹ , O-1126-95 |
| 24. Dicamba | GC | 615 | | | Note ³ , p. 115 |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| 25. Dichlofenthion | GC | 622.1 | | | Note ⁴ , page 27; Note ⁶ , p. S73 |
| 26. Dichloran | GC | 608.2, 617 | 6630 B-2000 | | Note ³ , p. 7 |
| 27. Dicofol | GC | 617 | | | Note ⁴ , O-3104-83 |
| 28. Dieldrin | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | Note ¹¹ , O-1126-95 |
| 29. Dioxathion | GC | 614.1, 1657 | | | Note ⁴ , page 27; Note ⁶ , p. S73 |
| 30. Disulfoton | GC | 507, 614, 622, 1657 | | | Note ³ , p. 25; Note ⁶ p. S51 |
| | GC/MS | 525.2 | | | Note ¹¹ , O-1126-95 |
| 31. Diuron | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | 553 | | | Note ¹² , O-2060-01 |
| 32. Endosulfan I | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 625 ⁵ | 6410 B-2000 | | Note ¹³ , O-2002-01 |
| 33. Endosulfan II | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁸ , 3M0222 |
| | GC/MS | 625 ⁵ | 6410 B-2000 | | Note ¹³ , O-2002-01 |
| 34. Endosulfan Sulfate | GC | 608, 617 | 6630 C-2000 | | Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |

Table D
List of Approved Test Procedures for Pesticides ¹

| Parameter | Analytical Technology | EPA 2,7,10 | Standard Methods | ASTM | Other |
|------------------------|-----------------------|--------------------------------|----------------------------|---------------------------|--|
| 35. Endrin | GC | 505, 508, 608, 617, 1656 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 525.1, 525.2, 625 ⁵ | 6410 B-2000 | | |
| 36. Endrin aldehyde | GC | 608, 617 | 6630 C-2000 | | Note ⁸ , 3M0222 |
| | GC/MS | 625 | | | |
| 37. Ethion | GC | 614, 614.1, 1657 | | | Note ⁴ , page 27; Note ⁶ , p. S73 |
| | GC/MS | | | | Note ¹³ , O-2002-01 |
| 38. Fenuron | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| 39. Fenuron-TCA | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| 40. Heptachlor | GC | 505, 508, 608, 617, 1656 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 525.1, 525.2, 625 | 6410 B-2000 | | |
| 41. Heptachlor epoxide | GC | 608, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁶ , p. S73; Note ⁸ , 3M0222 |
| | GC/MS | 625 | 6410 B-2000 | | |
| 42. Isodrin | GC | 617 | 6630 B-2000 6630 C-2000 | | Note ⁴ , O-3104-83; Note ⁶ , p. S73 |
| | GC/MS | | | | |
| 43. Linuron | GC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | 553 | | | Note ¹² , O-2060-01 |
| | GC/MS | | | | Note ¹¹ , O-1126-95 |
| 44. Malathion | GC | 614, 1657 | 6630 B-2000 | | Note ³ , p. 25; Note ⁶ , p. S51 |
| | GC/MS | | | | Note ¹¹ , O-1126-95 |
| 45. Methiocarb | TLC | | | | Note ³ , p. 94; Note ⁶ , p. S60 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| 46. Methoxychlor | GC | 505, 508, 608.2, 617, 1656 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83; Note ⁸ , 3M0222 |
| | GC/MS | 525.1, 525.2 | | | Note ¹¹ , O-1126-95 |
| 47. Mexacarbate | TLC | | | | Note ³ , p. 94; Note ⁶ , p. S60 |
| | HPLC | 632 | | | |
| 48. Mirex | GC | 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3104-83 |
| | GC/MS | | | | |
| 49. Monuron | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| 50. Monuron-TCA | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |

Table D
List of Approved Test Procedures for Pesticides ¹

| Parameter | Analytical Technology | EPA 2,7,10 | Standard Methods | ASTM | Other |
|-----------------------|-----------------------|---------------------|----------------------------|---------------------------|--|
| 51. Neburon | HPLC | 632 | | | |
| | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| 52. Parathion methyl | HPLC/MS | | | | Note ¹² , O-2060-01 |
| | GC | 614, 622, 1657 | 6630 B-2000 | | Note ³ , p. 25; Note ⁴ , page 27 |
| | GC/MS | | | | Note ¹¹ , O-1126-95 |
| 53. Parathion ethyl | GC | 614 | 6630 B-2000 | | Note ³ , p. 25; Note ⁴ , page 27 |
| | GC/MS | | | | Note ¹¹ , O-1126-95 |
| | | | | | |
| 54. PCNB | GC | 608.1, 617 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7. |
| 55. Perthane | GC | 617 | | D3086-90, D5812-96(02) | Note ⁴ , O-3104-83 |
| 56. Prometon | GC | 507, 619 | | | Note ³ , p. 83; Note ⁶ , p. S68; Note ⁹ , O-3106-93 |
| | GC/MS | 525.2 | | | Note ¹¹ , O-1126-95 |
| | | | | | |
| 57. Prometryn | GC | 507, 619 | | | Note ³ , p. 83; Note ⁶ , p. S68; Note ⁹ , O-3106-93 |
| | GC/MS | 525.1, 525.2 | | | Note ¹³ , O-2002-01 |
| | | | | | |
| 58. Propazine | GC | 507, 619, 1656 | | | Note ³ , p. 83; Note ⁶ , p. S68; Note ⁹ , O-3106-93 |
| | GC/MS | 525.1, 525.2. | | | |
| | | | | | |
| 59. Propham | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| 60. Propoxur | TLC | | | | Note ³ , p. 94; Note ⁶ , p. S60 |
| | HPLC | 632 | | | |
| | | | | | |
| 61. Sebumeton | TLC | | | | Note ³ , p. 83; Note ⁶ , p. S68 |
| | GC | 619 | | | |
| | | | | | |
| 62. Siduron | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| | HPLC/MS | | | | Note ¹² , O-2060-01 |
| 63. Simazine | GC | 505, 507, 619, 1656 | | | Note ³ , p. 83; Note ⁶ , p. S68; Note ⁹ , O-3106-93 |
| | GC/MS | 525.1, 525.2 | | | Note ¹¹ , O-1126-95 |
| | | | | | |
| 64. Strobane | GC | 617 | 6630 B-2000 6630 C-2000 | | Note ³ , p. 7 |
| 65. Swep | TLC | | | | Note ³ , p. 104; Note ⁶ , p. S64 |
| | HPLC | 632 | | | |
| | | | | | |
| 66. 2,4,5-T | GC | 615 | 6640 B-2001 | | Note ³ , p. 115; Note ⁴ , O-3105-83 |
| 67. 2,4,5-TP (Silvex) | GC | 615 | 6640 B-2001 | | Note ³ , p. 115; Note ⁴ , O-3105-83 |

Table D
List of Approved Test Procedures for Pesticides ¹

| Parameter | Analytical Technology | EPA ^{2,7,10} | Standard Methods | ASTM | Other |
|-------------------|-----------------------|--------------------------|----------------------------|---------------------------|--|
| 68. Terbutylazine | GC | 619, 1656 | | | Note ³ , p. 83; Note ⁶ , p. S68 |
| | GC/MS | | | | Note ¹³ , O-2002-01 |
| 69. Toxaphene | GC | 505, 508, 608, 617, 1656 | 6630 B-2000 6630 C-2000 | D3086-90, D5812-96(02) | Note ³ , p. 7; Note ⁴ , O-3105-83 Note ⁸ , 3M0222 |
| | GC/MS | 525.1, 525.2, 625 | 6410 B-2000 | | |
| 70. Trifluralin | GC | 508, 617, 627, 1656 | 6630 B-2000 | | Note ³ , p. 7; Note ⁹ , O-3106-93 |
| | GC/MS | 525.2 | | | Note ¹¹ , O-1126-95 |

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

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

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

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

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

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

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

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

⁹ Method O-3106-93 is in Open File Report 94-37, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-Containing Compounds by Gas Chromatography With Nitrogen Phosphorus Detectors. 1994. USGS.

¹⁰ EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, EPA 821-R-92-002, April 1992, U.S. EPA.

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

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

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

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

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

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

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

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

Table E
List of Approved Radiological Analytical Methods for Wastewater

| 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 |
| 4. Beta-Counting error, pCi | Proportional counter | Appendix B | 7110 B | 7110 B-00 | D1890-90, 96 | p. 79 |
| 5. (a) Radium Total pCi per liter | Proportional counter | 903.0 | 7500-Ra B | 7500-Ra B-01 | D2460-90, 97 | |
| (b) Radium, pCi per liter | Scintillation counter | 903.1 | 7500-Ra C | 7500-Ra C-01 | D3454-91, 97 | p. 81 |

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

² Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

³ The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

Table EM
List of Approved Analytical Methods for Sludge

| Parameter | Analytical Technology | Sample Preparation | | Determinative Method | | | |
|-----------|--|--------------------|------------------|----------------------|--------------------|------------------------------|-------|
| | | SW-8461 | EPA ⁴ | SW-846 ¹ | EPA ^{2,3} | Standard Methods [ed.] 8,9 | Other |
| Metals | Arsenic | | | | | | |
| | Gaseous Hydride ⁵ | 7061A | | 7061A | | | |
| | Graphite Furnace Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Beryllium | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | 3111 D [18,19,21], 3111 D-99 | |
| | Graphite Furnace Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |

Table EM
List of Approved Analytical Methods for Sludge

| Parameter | Analytical Technology | Sample Preparation | | Determinative Method | | | Other |
|------------|---|---------------------|------------------|----------------------|--------------------|--|-------|
| | | SW-846 ¹ | EPA ⁴ | SW-846 ¹ | EPA ^{2,3} | Standard Methods [ed.] 8,9 | |
| Cadmium | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | | |
| | Graphite Furnace Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Chromium | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | 3111 B [18,19,21], 3111 B-99 | |
| | Graphite Furnace Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Copper | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | 3111 B or C [18,19,21], 3111 B-99 or C-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Lead | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | | |
| | Graphite Furnace ⁶ Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Mercury | Cold Vapor Atomic Absorption | 7471A, 7471B | | 7471A, 7471B | | | |
| | Cold vapor atomic Fluorescence Spectrometry | 7474 | | | | | |
| Molybdenum | Graphite Furnace ⁶ Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |

Table EM
List of Approved Analytical Methods for Sludge

| Parameter | Analytical Technology | Sample Preparation | | Determinative Method | | | Other |
|-----------------------------------|--|--------------------------------|---------------------|------------------------------|---------------------------------|--|---|
| | | SW-846 ¹ | EPA ⁴ | SW-846 ¹ | EPA ^{2,3} | Standard Methods [ed.] 8,9 | |
| | Inductively Coupled Plasma/ Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Nickel | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | 3111 B or C [18,19,21], 3111 B-99 or C-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/ Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Selenium | Gaseous Hydride ⁵ | 7741A | | 7741A | | | |
| | Graphite Furnace Atomic Absorption | 3050B, 3051A | 200.2 | 7010 | 200.9 | 3113 B [18,19,21], 3113 B-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/ Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Zinc | Flame Atomic Absorption | 3050B, 3051A | 200.2 | 7000B | | 3111 B or C [18,19,21], 3111 B-99 or C-99 | |
| | Inductively Coupled Plasma Emission | 3050B, 3051A | 200.2 | 6010B, 6010C | 200.7 | 3120 B [20,21], 3120 B-99 | |
| | Inductively Coupled Plasma/ Mass Spectrometry | 3050B, 3051A | | 6020A | 200.8 | | |
| Organics | | | | | | | |
| Dioxins and Furans | Gas Chromatography/ Mass Spectrometry | 8290A ¹¹ | 1613B ¹¹ | 8290A | 1613B | | |
| PCBs (Aroclor or Congeners) | Gas Chromatography | 3540B, 3540C, 3545A | | 8082, 8082A ¹² | | | |
| PCB (Congeners) | Gas Chromatography/ Mass Spectrometry | 1668A ¹³ , 14,15 | | | 1668A ¹³ , 14, 15 | | |
| Biological | | | | | | | |
| Enteric Viruses | Centrifuge Concentration | | | | | | D 4994-89 (02) ⁷ , Appendix H ¹⁰ |

Table EM
List of Approved Analytical Methods for Sludge

| Parameter | Analytical Technology | Sample Preparation | | Determinative Method | | | Other |
|------------------------------------|---|---------------------|------------------|----------------------|--------------------|--|--|
| | | SW-846 ¹ | EPA ⁴ | SW-846 ¹ | EPA ^{2,3} | Standard Methods [ed.] 8,9 | |
| Fecal Coliform | Most Probable Number Membrane Filter | | | | | 9221 E [18,19, 20,21], 9221 E-99, 9222 D, 9222 D-97 | Appendix F ¹⁰ |
| Helminth ova | Density Gradient Flotation | | | | | | Note ⁹ or Appendix I ⁹ |
| Specific Oxygen Uptake Rate | Respirometer | | | | | 2710 B [18,19, 20,21], 2710 B-04 | Appendix D.2. ¹⁰ |
| Salmonella | Most Probable Number | | | | | | 9260 D.1 ⁸ |
| | Selective Media Culture | | | | | | Appendix G ¹⁰ |
| Physical | | | | | | | |
| Solids | Gravimetric | | | | | 2540 G [18,19,20,21], 2540 G-97 | |
| Percent Volatiles Solids Reduction | Calculation | | | | | | Appendix D.1. and 3 ¹⁰ |

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

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

³ "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.

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

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

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

⁷ 1993 Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993, 1916 Race Street, Philadelphia, PA 19103. Available from: the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

⁸ "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

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

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

- ¹¹ “Environmental Regulations and Technology – Control of Pathogens and Vectors Attraction in Sewage Sludge”, EPA-625/R-92/013, Revised October 1999, Environmental Protection Agency, Cincinnati, OH, 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.
- ¹² Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.
- ¹³ EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non-detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration by dry weight reported. It is recognized that a number of the congeners will co-elute with others, so there will not be 209 results to sum.
- ¹⁴ EPA Method 8082A shall be used for PCB-Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular sample. For either type of analysis, the sample shall be extracted using Soxhlet extraction Method 3540C or Pressurized Fluid Extraction Method 3545A. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and achieve as close to a limit of detection of 0.11 mg/kg as possible. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and to achieve as close to a limit of detection of 0.003 mg/kg as possible for each congener. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:
- 3620C – Florisil
 - 3640A – Gel Permeation
 - 3630C – Silica Gel
 - 3611B – Alumina
 - 3660B – Sulfur Clean Up
 - 3665A – Sulfuric Acid Clean Up.
- ¹⁵ “Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS”, EPA-821-R-00-002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Table ES
List of Approved Methods for Pharmaceutical Pollutants

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

¹ For compounds that also appear in Table C, test methods listed in Table C may also be used.

² EPA Methods 1666, 1667, and 1671 listed in the table above are published in the compendium titled Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters (EPA 821-B-98-016).

EPA Methods 502.2 and 524.2 have been incorporated by reference into 40 CFR 141.24 and are in Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, and Methods for the Determination of Organic Compounds in Drinking Water-Supplement II, EPA-600/R-92-129, August 1992, respectively. These EPA test method compendia are available from the National Technical Information Service, NTIS PB91-231480 and PB92-207703, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll-free number is 800-553-6847. ASTM test methods D3371, D3695, and D4763 are available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

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

Table F
Required Containers, Preservation Techniques, and Holding Times for wastewater

| Parameter Number/Name | Container ¹ | Preservation ^{2,3} | Maximum Holding Time ⁴ |
|--|------------------------|---|-----------------------------------|
| Table A — Bacterial Tests | | | |
| 1-5. Coliform, total, fecal and <i>E. coli</i> | PA, G | Cool, <10°C, 0.0008% Na ₂ S ₂ O ₃ ⁵ | 8 hours. ^{22,23} |
| 6. Fecal streptococci | PA, G | Cool, <10°C, 0.0008% Na ₂ S ₂ O ₃ ⁵ | 8 hours. ²² |
| 7. Enterococci | PA, G | Cool, <10°C, 0.0008% Na ₂ S ₂ O ₃ ⁵ | 8 hours. ²² |
| 8. <i>Salmonella</i> | PA, G | Cool, <10°C, 0.0008% Na ₂ S ₂ O ₃ ⁵ | 8 hours. ²² |
| Table A — Aquatic Toxicity Tests | | | |
| 9-12. Toxicity, acute and chronic | P, FP, G | Cool, ≤6°C ¹⁶ | 36 hours |

Table F (Continued)
Required Containers, Preservation Techniques, and Holding Times for wastewater

| Parameter Number/Name | Container ¹ | Preservation ^{2,3} | Maximum Holding Time ⁴ |
|---|------------------------|--|--|
| Table B — Inorganic Tests | | | |
| 1. Acidity | P, FP, G | Cool, ≤6°C ¹⁸ | 14 days |
| 2. Alkalinity | P, FP, G | Cool, ≤6°C ¹⁸ | 14 days |
| 4. Ammonia | P, FP, G | Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2 | 28 days |
| 9. Biochemical oxygen demand | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |
| 11. Bromide | P, FP, G | None required | 28 days |
| 14. 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 ₂ SO ₄ to pH<2 | 28 days |
| 16. Chloride | P, FP, G | None required | 28 days |
| 17. Chlorine, total residual | P, G | None required | Analyze within 15 minutes |
| 21. Color | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |
| 23-24. Cyanide, total or available (or CATC) free | P, FP, G | Cool, ≤6°C ¹⁸ , NaOH to pH>10 ⁶ , reducing agent if oxidizer present | 14 days |
| 25. Fluoride | P | None required | 28 days |
| 27. Hardness | P, FP, G | HNO ₃ or H ₂ SO 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 ₂ SO ₄ to pH<2 | 28 days |
| 38. Nitrate | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |
| 39. Nitrate – nitrite | P, FP, G | Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2 | 28 days |
| 40. Nitrite | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |
| 41. Oil and grease | G | Cool, ≤6°C ¹⁸ , HCl or H ₂ SO ₄ to pH<2 | 28 days |
| 42. Organic carbon | P, FP, G | Cool, ≤6°C ¹⁸ , HCl, H ₂ SO ₄ or H ₃ PO ₄ to pH<2 | 28 days |
| 44. Orthophosphate | P, FP, G | Cool, to ≤6°C ^{18,24} | Filter within 15 minutes; Analyze within 48 hours. |
| 46. Oxygen, dissolved (Probe or Luminescence) | G, Bottle and top | None required | Analyze within 15 minutes |
| 47. Oxygen, Dissolved Winkler | G, Bottle and top | Fix on site and store in dark | 8 hours |
| 48. Phenols | G | Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2 | 28 days |
| 49. Phosphorus (elemental) | G | Cool, ≤6°C ¹⁸ | 48 hours |
| 50. Phosphorus, total | P, FP, G | Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH<2 | 28 days |
| 53. Residue, total | P, FP, G | Cool, ≤6°C ¹⁸ | 7 days |
| 54. Residue, Filterable (TDS) | P, FP, G | Cool, ≤6°C ¹⁸ | 7 days |
| 55. Residue, Nonfilterable (TSS) | P, FP, G | Cool, ≤6°C ¹⁸ | 7 days |
| 56. Residue, Settleable | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |
| 57. Residue, Volatile | P, FP, G | Cool, ≤6°C ¹⁸ | 7 days |
| 61. Silica | P or Quartz | Cool, ≤6°C ¹⁸ | 28 days |
| 64. Specific conductance | P, FP, G | Cool, ≤6°C ¹⁸ | 28 days |
| 65. Sulfate | P, FP, G | Cool, ≤6°C ¹⁸ | 28 days |
| 66. Sulfide | P, FP, G | Cool, ≤6°C ¹⁸ , add zinc acetate plus sodium hydroxide to pH>9 | 7 days |
| 67. Sulfite | P, FP, G | None required | Analyze within 15 minutes |
| 68. Surfactants | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |
| 69. Temperature | P, FP, G | None required | Analyze |
| 73. Turbidity | P, FP, G | Cool, ≤6°C ¹⁸ | 48 hours |

Table F (Continued)
Required Containers, Preservation Techniques, and Holding Times for wastewater

| Parameter Number/Name | Container ¹ | Preservation ^{2,3} | Maximum Holding Time ⁴ |
|--|---------------------------------------|---|---|
| Table B — Metals⁷ | | | |
| 10. Boron | P, FP, or Quartz | HNO ₃ to pH<2 | 6 months |
| 18. Chromium VI | P, FP, G | Cool, ≤6°C ¹⁸ , pH = 9.3 – 9.7 ²⁰ | 28 days |
| 35. Mercury (CVAA) | P, FP, G | HNO ₃ to pH<2 | 28 days |
| 35. Mercury (CVAFS) | FP, G; and FP-lined cap ¹⁷ | 5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷ | 90 days ¹⁷ |
| 3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except boron, chromium VI, and mercury. | P, FP, G | HNO ₃ to pH<2, or at least 24 hours prior to analysis ¹⁹ | 6 months |
| Table C — Organic Tests⁸ | | | |
| 3, 4. Acrolein and acrylonitrile | G, FP-lined septum | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ , pH to 4-5 ¹⁰ | 14 days ¹⁰ |
| 119. Adsorbable Organic Halides (AOX) | G | Cool, <6°C, 0.008% Na ₂ S ₂ O ₃ HNO ₃ to pH <2 | Hold <i>at least</i> 3 days, but not more than 6 months |
| 114-118. Alkylated phenols | G | Cool, <6°C, H ₂ SO ₄ to pH <2 | 28 days until extraction, 40 days after extraction |
| 7, 38. Benzidines ^{11, 12} | G, FP-lined cap | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ | 7 days until extraction ¹³ |
| 29, 35-37, 63-65, 107. Chlorinated hydrocarbons ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ | 7 days until extraction, 40 days after extraction |
| 120. Chlorinated Phenolics | | Cool, <6°C, 0.008% Na ₂ S ₂ O ₃ , H ₂ SO ₄ to pH <2 | 30 days until acetylation, 30 days after acetylation. |
| 15, 16, 21, 31, 87. Haloethers ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ | 7 days until extraction, 40 days after extraction |
| 54, 55, 75, 79. Nitroaromatics and Isophorone ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ | 7 days until extraction, 40 days after extraction |
| 82-84. Nitrosamines ^{11, 14} | G, FP-lined cap | Cool, ≤6°C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ | 7 days until extraction, 40 days after extraction |
| 88-94. PCBs ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ | 1 year until extraction, 1 year after extraction |
| 60-62, 66-72, 85, 86, 95-97, 102, 103. PCDDs/PCDFs ¹¹ | | | |
| Aqueous Samples: Field and Lab Preservation | G | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O 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 |
| 23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ | 7 days until extraction, 40 days after extraction |
| 14, 17, 48, 50-52. Phthalate esters ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ | 7 days until extraction, 40 days after extraction |
| 1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹¹ | G, FP-lined cap | Cool, ≤6°C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ | 7 days until extraction, 40 days after extraction |
| 6, 57, 106. Purgeable aromatic hydrocarbons | G, FP-lined septum | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹ | 14 days ⁹ |
| 13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable halocarbons. | G, FP-lined septum | Cool, ≤6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ | 14 days |

Table F (Continued)
Required Containers, Preservation Techniques, and Holding Times for wastewater

| Parameter Number/Name | Container ¹ | Preservation ^{2,3} | Maximum Holding Time ⁴ |
|--------------------------------------|------------------------|--|--|
| Table D — Pesticides Tests: | | | |
| 1-70. Pesticides ¹¹ | G, FP-lined cap | Cool, ≤ 6°C ¹⁸ , pH 5-9 ¹⁵ | 7 days until extraction, 40 days after extraction |
| Table E — Radiological Tests: | | | |
| 1-5. Alpha, beta and radium | P, FP, G | HNO ₃ to pH<2 | 6 months |
| Table H — Bacterial Tests: | | | |
| 1. <i>E. coli</i> | PA, G | Cool, <10°C, 0.0008% Na ₂ S ₂ O ₃ ⁵ | 8 hours. ²² |
| 2. Enterococci | PA, G | Cool, <10°C, 0.0008% Na ₂ S ₂ O ₃ ⁵ | 8 hours. ²² |
| Table H — Protozoan Tests: | | | |
| 8. <i>Cryptosporidium</i> | LDPE; field filtration | 1-10 °C | 96 hours. ²¹ |
| 9. <i>Giardia</i> | LDPE; field filtration | 1-10 °C | 96 hours. ²¹ |

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

² Except where noted in this table and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (e.g., using a 24-hour composite sampler, refrigerate the sample at ≤ 6 °C during collection unless specified otherwise in this table or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤ 6 °C, unless specified otherwise in this table or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection.

The temperature of the samples shall be documented upon receipt at the laboratory. If the samples are shipped in crushed or cube ice (not “blue ice” packs) and solid ice is still present in the cooler, the lab may simply report the samples as “received on ice”. If the ice has melted, the lab must report the either the temperature of the melt- water or of a temperature blank. A temperature blank is defined as an aliquot of deionized water, in an appropriate sample container, which is transported along with the samples. Since shipping simply with “blue ice” packs does not insure that samples are maintained at the appropriate temperatures, the sample collector must submit a temperature blank when using these ice packs for shipping.

³ When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirement, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

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

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

⁶ Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to >12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to >12 and refrigeration as specified.

There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH >12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

Sulfur: To remove elemental sulfur (S8), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to >12 with NaOH,

refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH–extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in μg or mg), and divide by the original sample volume to obtain the cyanide concentration.

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

Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH–extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in g or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand–exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand exchange reagent to offset any excess of cadmium chloride.

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

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

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

(5) Chlorine, hypochlorite, or other oxidant: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5⁷ For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler, filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

⁷ For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler, filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

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

⁹ If the sample is not adjusted to pH < 2, then the sample must be analyzed within seven days of sampling.

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

¹¹ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (*i.e.*, use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to $\leq 6^\circ\text{C}$, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

- ¹² If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.
- ¹³ Extracts may be stored up to 30 days at < 0 °C.
- ¹⁴ For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- ¹⁵ The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.
- ¹⁶ Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.
- ¹⁷ Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
- ¹⁸ Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of " ≤ 6 °C" is used in place of the "4 °C" and " < 4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤ 6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
- ¹⁹ An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.
- ²⁰ To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.
- ²¹ Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.
- ²² Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.
- ²³ For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB-EC) or 1681 (A-1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.
- ²⁴ The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (*i.e.*, that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (*i.e.*, within 15 minutes of collection).

Table G
Test Methods for Pesticide Active Ingredients

| EPA survey code | Pesticide name | CAS No. | EPA analytical method No.(s) ³ |
|-----------------|---|------------|---|
| 8 | Triadimefon | 43121-43-3 | 1656, 507, 633, 525.1, 525.2 |
| 12 | Dichlorvos | 62-73-7 | 1657, 507, 525.1, 525.2, 622 |
| 16 | 2,4-D; 2,4-D Salts and Esters [2,4-Dichloro-phenoxyacetic acid] | 94-75-7 | 1658, 515.1, 515.2, 555, 615 |
| 17 | 2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid] | 94-82-6 | 1658, 515.1, 515.2, 555, 615 |
| 22 | Mevinphos | 7786-34-7 | 1657, 507, 525.1, 525.2, 622 |
| 25 | Cyanazine | 21725-46-2 | 507, 629 |
| 26 | Propachlor | 1918-16-7 | 1656, 508, 608.1, 525.1, 525.2 |
| 27 | MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid] | 94-74-6 | 1658, 555, 615 |
| 30 | Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid] | 120-36-5 | 1658, 515.1, 515.2, 555, 615 |
| 31 | MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid] | 93-65-2 | 1658, 555, 615 |

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Test Methods for Pesticide Active Ingredients

| EPA survey code | Pesticide name | CAS No. | EPA analytical method No.(s) ³ |
|-----------------|--|------------|--|
| 35 | TCMTB [2-(Thiocyanomethylthio) benzo-thiazole] | 21564-17-0 | 637 |
| 39 | Pronamide | 23950-58-5 | 507, 525.1, 525.2, 633.1 |
| 41 | Propanil | 709-98-8 | 1656, 632.1 |
| 45 | Metribuzin | 21087-64-9 | 1656, 507, 525.1, 525.2, 633 |
| 52 | Acephate | 30560-19-1 | 1656, 1657 |
| 53 | Acifluorfen | 50594-66-6 | 515.1, 515.2, 555 |
| 54 | Alachlor | 15972-60-8 | 1656, 505, 507, 525.1, 525.2, 645 |
| 55 | Aldicarb | 116-06-3 | 531.1 |
| 58 | Ametryn | 834-12-8 | 507, 525.2, 619 |
| 60 | Atrazine | 1912-24-9 | 1656, 505, 507, 525.1, 525.2, 619 |
| 62 | Benomyl | 17804-35-2 | 631 |
| 68 | Bromacil; Bromacil Salts and Esters | 314-40-9 | 1656, 507, 525.1, 525.2, 633 |
| 69 | Bromoxynil | 1689-84-5 | 1625, 1661 |
| 69 | Bromoxynil octanoate | 1689-99-2 | 1656 |
| 70 | Butachlor | 23184-66-9 | 1656, 507, 525.1, 525.2, 645 |
| 73 | Captafol | 2425-06-1 | 1656 |
| 75 | Carbaryl [Sevin] | 63-25-2 | 531.1, 553, 632 |
| 76 | Carbofuran | 1563-66-2 | 531.1, 632 |
| 80 | Chloroneb | 2675-77-6 | 1656, 508, 525.1, 525.2, 608.1 |
| 82 | Chlorothalonil | 1897-45-6 | 1656, 508, 525.1, 525.2, 608.2 |
| 84 | Stirofos | 961-11-5 | 1657, 507, 525.1, 525.2, 622 |
| 86 | Chlorpyrifos | 2921-88-2 | 1657, 508, 622 |
| 90 | Fenvalerate | 51630-58-1 | 1660 |
| 103 | Diazinon | 333-41-5 | 1657, 507, 525.2, 614, 622 |
| 107 | Parathion methyl | 298-00-0 | 1657, 614, 622 |
| 110 | DCPA [Dimethyl 2,3,5,6-tetrachloro-terephthalate] | 1861-32-1 | 1656, 508, 525.1, 525.2, 515.1 ² , 515.2 ² , 608.2 |
| 112 | Dinoseb | 88-85-7 | 1658, 515.1, 515.2, 555, 615 |
| 113 | Dioxathion | 78-34-2 | 1657, 614.1 |
| 118 | Nabonate [Disodium cyanodithioimidocarbonate] | 138-93-2 | 630.1 |
| 119 | Diuron | 330-54-1 | 553, 632 |
| 123 | Endothall | 145-73-3 | 548, 548.1 |
| 124 | Endrin | 72-20-8 | 1656, 505, 508, 525.1, 525.2, 608, 617 |
| 125 | Ethalfuralin | 55283-68-6 | 1656, 627 See footnote 1 |
| 126 | Ethion | 563-12-2 | 1657, 614, 614.1 |
| 127 | Ethoprop | 13194-48-4 | 1657, 507, 525.1, 525.2, 622 |
| 132 | Fenarimol | 60168-88-9 | 1656, 507, 525.1, 525.2, 633.1 |
| 133 | Fenthion | 55-38-9 | 1657, 622 |
| 138 | Glyphosate [N-(Phosphonomethyl) glycine] | 1071-83-6 | 547 |
| 140 | Heptachlor | 76-44-8 | 1656, 505, 508, 525.1, 525.2, 608, 617 |
| 144 | Isopropalin | 33820-53-0 | 1656, 627 |
| 148 | Linuron | 330-55-2 | 553, 632 |
| 150 | Malathion | 121-75-5 | 1657, 614 |
| 154 | Methamidophos | 10265-92-6 | 1657 |
| 156 | Methomyl | 16752-77-5 | 531.1, 632 |
| 158 | Methoxychlor | 72-43-5 | 1656, 505, 508, 525.1, 525.2, 608.2, 617 |
| 172 | Nabam | 142-59-6 | 630, 630.1 |
| 173 | Naled | 300-76-5 | 1657, 622 |
| 175 | Norflurazon | 27314-13-2 | 1656, 507, 525.1, 525.2, 645 |
| 178 | Benfluralin | 1861-40-1 | 1656, 627 See footnote 1 |

Table G
Test Methods for Pesticide Active Ingredients

| EPA survey code | Pesticide name | CAS No. | EPA analytical method No.(s) ³ |
|-----------------|--|------------|--|
| 182 | Fensulfothion | 115-90-2 | 1657, 622 |
| 183 | Disulfoton | 298-04-4 | 1657, 507, 525.2, 614, 622 |
| 185 | Phosmet | 732-11-6 | 1657, 622.1 |
| 186 | Azinphos Methyl | 86-50-0 | 1657, 614, 622 |
| 192 | Organo-tin pesticides | 12379-54-3 | 200.7, 200.9, Ind-01 |
| 197 | Bolstar | 35400-43-2 | 1657, 622 |
| 203 | Parathion | 56-38-2 | 1657, 614 |
| 204 | Pendimethalin | 40487-42-1 | 1656 |
| 205 | Pentachloronitrobenzene | 82-68-8 | 1656, 608.1, 617 |
| 206 | Pentachlorophenol | 87-86-5 | 1625, 515.2, 555, 515.1, 525.1, 525.2, 625 |
| 208 | Permethrin | 52645-53-1 | 1656, 1660, 508, 525.1, 525.2, 608.2 |
| 212 | Phorate | 298-02-2 | 1657, 622 |
| 218 | Busan 85 [Potassium dimethyldithiocarbamate] | 128-03-0 | 630, 630.1 |
| 219 | Busan 40 [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, 525.2, 619 |
| 224 | Prometryn | 7287-19-6 | 507, 525.1, 525.2, 619 |
| 226 | Propazine | 139-40-2 | 1656, 507, 525.1, 525.2, 619 |
| 230 | Pyrethrin I | 121-21-1 | 1660 |
| 232 | Pyrethrin II | 121-29-9 | 1660 |
| 236 | DEF [S,S,S-Tributyl phosphorotrithioate] | 78-48-8 | 1657 |
| 239 | Simazine | 122-34-9 | 1656, 505, 507, 525.1, 525.2, 619 |
| 241 | Carbam-S [Sodium dimethyldithio-carbamate] | 128-04-1 | 630, 630.1 |
| 243 | Vapam [Sodium methyldithiocarbamate] | 137-42-8 | 630, 630.1 |
| 252 | Tebuthiuron | 34014-18-1 | 507, 525.1, 525.2 |
| 254 | Terbacil | 5902-51-2 | 1656, 507, 525.1, 525.2, 633 |
| 255 | Terbufos | 13071-79-9 | 1657, 507, 614.1, 525.1, 525.2 |
| 256 | Terbutylazine | 5915-41-3 | 1656, 619 |
| 257 | Terbutryn | 886-50-0 | 507, 525.1, 525.2, 619 |
| 259 | Dazomet | 533-74-4 | 1659, 630, 630.1 |
| 262 | Toxaphene | 8001-35-2 | 1656, 505, 508, 525.1, 525.2, 608, 617 |
| 263 | Merphos [Tributyl phosphorotrithioate] | 150-50-5 | 1657, 507, 525.1, 525.2, 622 |
| 264 | Trifluralin ¹ | 1582-09-8 | 1656, 508, 525.2, 617, 627 |
| 268 | Ziram [Zinc dimethyldithiocarbamate] | 137-30-4 | 630, 630.1 |

¹ Monitor and report as total Trifluralin.

² Applicable to the analysis of DCPA degradates.

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

Table H
List of Approved Microbiological Methods for Ambient Water

| Parameter and units | Method ¹ | EPA | Standard methods | AOAC, ASTM, USGS | Other |
|--|---|--|--|------------------------|--|
| Bacteria: | | | | | |
| 1. Coliform (fecal), number per 100 mL or number per gram dry weight | Most Probable Number (MPN), 5 tube, 3 dilution, or | p. 132 ³ | 9221 C E-2006 | | |
| | Membrane filter (MF) ² , single step | p. 124 ³ | 9222 D-1997 | B-0050-85 ⁴ | |
| 2. Coliform (fecal) in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 132 ³ | 9221 C, E-2006 | | |
| | MF ² , single step ⁵ | p. 124 ³ | 9222 D-1997 | | |
| 3. Coliform (total), number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 ³ | 9221 B-2006 | | |
| | MF ² , single step or two step | p. 108 ³ | 9222 B-1997 | B-0025-85 ⁴ | |
| 4. Coliform (total), in presence of chlorine, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 114 ³ | 9221 B-2006 | | |
| | MF ² with enrichment | p. 111 ³ | 9222 (B+B.5c)-1997 | | |
| 5. <i>E. coli</i> , number per 100 mL | MPN ^{6, 8, 14} , multiple tube, or | | 9221 B.1-2006/ 9221 F-2006 ^{11, 13} | | |
| | Multiple tube/multiple well, or | | 9223 B-2004 ¹² | 991.15 ¹⁰ | Colilert [®] ^{12, 16} Colilert-18 [®] ^{12, 15, 16} |
| | MF ^{2, 5, 6, 7, 8} , two step, or | 1103.1 ¹⁹ | 9222 B-1997/ 9222 G-1997 ¹⁸ 9213 D-2007 | D5392-93 ⁹ | |
| | Single step | 1603 ²⁰ , 1604 ²¹ | | | mColiBlue-24 ^{®17} |
| 6. Fecal streptococci, number per 100 mL | MPN, 5 tube, 3 dilution, or | p. 139 ³ | 9230 B-2007 | | |
| | MF ² , or | p. 136 ³ | 9230 C-2007 | B-0055-85 ⁴ | |
| | Plate count | p. 143 ³ | | | |
| 7. Enterococci, number per 100 mL | MPN ^{6, 8} , multiple tube/multiple well, or | | | D6503-99 ⁹ | Enterolert [®] ^{12, 22} |
| | MF ^{2, 5, 6, 7, 8} two step, or | 1106.1 ²³ | 9230 C-2007 | D5259-92 ⁹ | |
| | Single step, or | 1600 ²⁴ | 9230 C-2007 | | |
| | Plate count | p. 143 ³ | | | |
| Protozoa: | | | | | |
| 8. <i>Cryptosporidium</i> | Filtration/IMS/FA | 1622 ²⁵ 1623 ²⁶ | | | |
| 9. <i>Giardia</i> | Filtration/IMS/FA | 1623 ²⁶ | | | |

¹ The method must be specified when results are reported.

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

³ Microbiological Methods for Monitoring the Environment, Water, and Wastes. EPA/600/8-78/017. 1978. US EPA.

⁴ U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. USGS.

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

⁶ Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

⁷ When the MF method has not been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

⁸ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

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

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

- ¹¹ The multiple–tube fermentation test is used in 9221B.1–2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false–positive rate and false–negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform–positive tubes on a seasonal basis.
- ¹² These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β –glucuronidase produced by *E. coli*.
- ¹³ After prior enrichment in a presumptive medium for total coliform using 9221B.1–2006, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h \pm 3 h of incubation shall be submitted to 9221F–2006. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 μ g/mL of MUG may be used.
- ¹⁴ Samples shall be enumerated by the multiple–tube or multiple–well procedure. Using multiple–tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple–well procedures, Quanti–Tray® or Quanti–Tray®/2000, and the MPN calculated from the table provided by the manufacturer.
- ¹⁵ Colilert–18® is an optimized formulation of the Colilert® for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C, rather than the 24 h required for the Colilert® test, and is recommended for marine water samples.
- ¹⁶ Descriptions of the Colilert®, Colilert–18®, Quanti–Tray®, and Quanti–Tray®/2000 may be obtained from IDEXX Laboratories Inc.
- ¹⁷ A description of the mColiBlue24® test may be obtained from Hach Company.
- ¹⁸ Subject total coliform positive samples determined by 9222B–1997 or other membrane filter procedure to 9222G–1997 using NA–MUG media.
- ¹⁹ Method 1103.1: *Escherichia coli*(*E. coli*) in Water by Membrane Filtration Using membrane–Thermotolerant *Escherichia coli* Agar (mTEC), EPA–821–R–10–002. March 2010. US EPA.
- ²⁰ Method 1603: *Escherichia coli*(*E. coli*) in Water by Membrane Filtration Using Modified membrane–Thermotolerant *Escherichia coli* Agar (Modified mTEC), EPA–821–R–09–007. December 2009. US EPA.
- ²¹ Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. New Medium for the Simultaneous Detection of Total Coliform and *Escherichia coli* in Water. Appl. Environ. Microbiol. 59:3534–3544 and in Method 1604: Total Coliforms and *Escherichia coli*(*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium), EPA 821–R–02–024, September 2002, US EPA.
- ²² A description of the Enterolert® test may be obtained from IDEXX Laboratories Inc.
- ²³ Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane–Enterococcus–Esculin Iron Agar (mE–EIA), EPA–821–R–09–015. December 2009. US EPA.
- ²⁴ Method 1600: Enterococci in Water by Membrane Filtration Using membrane–Enterococcus Indoxyl– β –D–Glucoside Agar (mEI), EPA–821–R–09–016. December 2009. US EPA.
- ²⁵ Method 1622 uses a filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA, EPA–821–R–05–001. December 2005. US EPA.
- ²⁶ Method 1623 uses a filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. EPA–821–R–05–002. December 2005. US EPA.