ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[EPA–HQ–OW–2018–0826; FRL–9995–22–OW]

RIN 2040–AF84

Clean Water Act Methods Update Rule for the Analysis of Effluent

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule.

SUMMARY: The Environmental Protection Agency (EPA) is proposing changes to its test procedures required to be used by industries and municipalities when analyzing the chemical, physical, and biological properties of wastewater and other environmental samples for reporting under the EPA's National Pollutant Discharge Elimination System (NPDES) permit program. The Clean Water Act requires the EPA to promulgate these test procedures (analytical methods) for analysis of pollutants. The EPA anticipates that these proposed changes will provide increased flexibility for the regulated community in meeting monitoring requirements while improving data quality. In addition, this proposed update to the CWA methods would incorporate technological advances in analytical technology. As such, the EPA expects that there will be no negative economic impacts resulting from these proposed changes.

DATES: Comments on this proposed rule must be received on or before December 23, 2019.

ADDRESSES: You may send comments, identified by Docket ID No. EPA–HQ–OW–2018–0826, by any of the following means:

- *Federal eRulemaking Portal: https://www.regulations.gov/_* (preferred way of receiving comments): Follow the online instructions for submitting comments.
- *Email: OW-Docket@epa.gov.* Include Docket ID No. EPA–HQ–OW–2018–0826 in the subject line of the message.
- *Mail:* U.S. Environmental Protection Agency, EPA Docket Center, Office of Water Docket, Mail Code 28221T, 1200 Pennsylvania Avenue NW, Washington, DC 20460.
- *Hand Delivery/Courier:* EPA Docket Center, WJC West Building, Room 3334, 1301 Constitution Avenue NW, Washington, DC 20004. The Docket Center's hours of operations are 8:30 a.m.–4:30 p.m., Monday–Friday (except Federal Holidays).

Instructions: All submissions received must include the Docket ID No. for this rulemaking. Comments received may be posted without change to *https://www.regulations.gov/,* including any personal information provided. For detailed instructions on sending comments and additional information on the rulemaking process, see Section I.B ''What Should I Consider as I Prepare My Comments for the EPA'' heading of the **SUPPLEMENTARY INFORMATION** section of this document.

Docket: All documents in the docket are listed in the *www.regulations.gov* index. Although listed in the index, some information in the docket is not publicly available, *e.g.,* Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, will be publicly available only in hard copy. Publicly available docket materials are available either electronically in *www.regulations.gov* or in hard copy at the Water Docket in EPA Docket Center, EPA/DC, EPA West William J. Clinton Building, Room 3334, 1301 Constitution Avenue NW, Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is 202–566–1744, and the telephone number for the Water Docket is 202–566–2426.

FOR FURTHER INFORMATION CONTACT: Meghan Hessenauer, Engineering and Analysis Division (4303T), Office of Water, Environmental Protection Agency, 1200 Pennsylvania Avenue NW, Washington, DC 20460–0001; telephone: 202–566–1040; email: *Hessenauer.Meghan@epa.gov.*

SUPPLEMENTARY INFORMATION:

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I. General Information

A. Does this action apply to me?

Entities potentially affected by the requirements of this proposed action include:

Category	Examples of potentially affected entities
State, Territorial, and Indian Tribal Governments.	States, territories, and tribes authorized to administer the National Pollutant Discharge Elimination System (NPDES) permitting program; states, territories, and tribes providing certification under CWA section 401; state, territorial, and tribal-owned facilities that must conduct monitoring to comply with NPDES permits.
Industry	Facilities that must conduct monitoring to comply with NPDES permits.
Municipalities	Publicly Owned Treatment Works (POTWs) or other municipality-owned facilities that must conduct monitoring to comply with NPDES permits.

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

B. What should I consider as I prepare my comments for the EPA?

Submit your comments, identified by Docket ID No. EPA–HQ–OW–2018–0826, at *https://www.regulations.gov* (preferred way of receiving comments), or the other means identified in the **ADDRESSES** section. Once submitted, comments cannot be edited or removed from the docket. The EPA may publish any comment received to its public docket. Do not submit electronically any information you consider to be CBI or other information whose disclosure is restricted by statute. Do not submit CBI to the EPA through *www.regulations.gov* or email. Clearly mark the part or all of the information that you claim to be CBI. For CBI information in a disk that you mail to the EPA, mark the outside of the disk as CBI and then identify electronically within the disk the specific information that is claimed as CBI. In addition to one complete version of the comment that includes information claimed as CBI, a copy of the comment that does not contain the information claimed as CBI must be submitted for inclusion in the public docket. Information so marked will not be disclosed except in accordance with procedures for handling and protection of CBI set forth in 40 CFR part 2.

Multimedia submissions (audio, video, etc.) must be accompanied by a written comment. The written comment is considered the official comment and should include discussion of all points you wish to make. The EPA will generally not consider comments or comment contents located outside of the primary submission (*i.e.,* on the web, cloud, or other file sharing system). For additional submission means or methods, the full EPA public comment policy, information about CBI or multimedia submissions, and general guidance on making effective comments, please visit *https://www.epa.gov/dockets/commenting-epa-dockets.*

II. Overview

This preamble describes the reasons for the proposed rule; the legal authority for the proposed rule; a summary of the proposed changes and clarifications; and explanation of the abbreviations and acronyms used in this document. In addition, this preamble solicits comment and data from the public.

Abbreviations and Acronyms Used in the Preamble and Proposed Rule Text

2-CEVE: 2-Chloroethylvinyl ether
AA: Atomic Absorption
ADMI: American Dye Manufacturers Institute
ASTM: ASTM International[1]
ATP: Alternate Test Procedure
BHI: Brain heart infusion
BOD_5: 5-day Biochemical Oxygen Demand
CAS: Chemical Abstract Services
CATC: Cyanide Amenable to Chlorination
CCB: Continuing calibration blank
CCV: Continuing calibration verification
CFR: Code of Federal Regulations
COD: Chemical Oxygen Demand
CWA: Clean Water Act
EC-MUG: EC broth with 4-methylumbelliferyl-β-D-glucuronide
EDTA: Ethylenediaminetetraacetic acid
ELAB: Environmental Laboratory Advisory Board
EPA: Environmental Protection Agency
FLAA: Flame Atomic Absorption Spectroscopy
GC: Gas Chromatography
GFAA: Graphite Furnace Atomic Absorption Spectroscopy
ICP/AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP/MS: Inductively Coupled Plasma-Mass Spectrometry
ILI: Independent Laboratories Institute
IPR: Initial Precision and Recovery
LCS: Laboratory Control Sample
MDL: Method Detection Limit
MF: Membrane Filtration
$MgCl_2$: Magnesium Chloride
MPN: Most Probable Number
MS/MSD: Matrix Spike/Matrix Spike Duplicate
MS: Mass Spectrometry
NA-MUG: Nutrient Agar with 4-methylumbelliferyl-β-D-glucuronide
NECi: A shortened name used by the Nitrate Elimination Company, Inc.
NPDES: National Pollutant Discharge Elimination System
NTTAA: National Technology Transfer and Advancement Act
OPR: Ongoing Precision and Recovery
QC: Quality Control
STGFAA: Stabilized Temperature Graphite Furnace Atomic Absorption Spectroscopy
TKN: Total Kjeldahl Nitrogen
TOC: Total Organic Carbon
USGS: United States Geological Survey
VCSB: Voluntary Consensus Standards Body

III. Statutory Authority

The EPA is proposing this regulation under the authorities of sections 301(a), 304(h), and 501(a) of the CWA; 33 U.S.C. 1311(a), 1314(h), and 1361(a). Section 301(a) of the CWA prohibits the discharge of any pollutant into navigable waters unless the discharge complies with, among other provisions, an NPDES permit issued under section 402 of the CWA. Section 304(h) of the CWA requires the Administrator of the EPA to ''. . . promulgate guidelines establishing test procedures for the analysis of pollutants that shall include the factors which must be provided in any certification pursuant to [section 401 of the CWA] or permit application pursuant to [section 402 of the CWA].'' Section 501(a) of the CWA authorizes the Administrator to ''. . . prescribe such regulations as are necessary to carry out this function under [the CWA].'' The EPA generally has codified its test procedure regulations (including analysis and sampling requirements) for CWA programs at 40 CFR part 136, though some requirements are codified in other parts (*e.g.,* 40 CFR Chapter I, Subchapters N and O).

IV. Purpose and Summary of Proposed Rule

NPDES permits must include conditions designed to ensure compliance with the technology-based and water quality-based requirements of the CWA, including in many cases, restrictions on the quantity of specific pollutants that can be discharged as well as pollutant measurement and reporting requirements. Often, entities have a choice in deciding which approved test procedure they will use for a specific pollutant because the EPA has approved the use of more than one method.[2]

The procedures for the analysis of pollutants required by CWA section 304(h) are a central element of the NPDES permit program. Examples of where these EPA-approved analytical methods must be used include the following: (1) Applications for NPDES permits, (2) sampling or other reports required under NPDES permits, (3) other requests for quantitative or qualitative effluent data under the NPDES regulations, (4) State CWA 401 certifications and (5) sampling and analysis required under the EPA's General Pretreatment Regulations for Existing and New Sources of Pollution, 40 CFR 136.1 and 40 CFR 403.12(b)(5)(v).

Periodically, the EPA proposes to update the approved methods in 40 CFR part 136. In general, the changes proposed in this action fall into the following categories. The first is new or revised methods published by the VCSBs or the USGS that are similar to methods previously adopted as EPA-approved methods in 40 CFR part 136. The second category is methods the EPA has reviewed under the Agency's national ATP program and preliminarily concluded are appropriate for nationwide use. Finally, the EPA is proposing certain corrections or amendments to the text and tables of 40 CFR part 136. The EPA is proposing adoption of these revisions to improve data quality, update methods to keep current with technology advances, and provide the regulated community with greater flexibility. The following paragraphs provide details on the proposed revisions.

A. Changes to 40 CFR 136.3 To Include New Versions of Previously Approved EPA Methods

The EPA proposes to add the latest version of EPA Method 1623 to Table IH. The latest version of Method 1623 (labeled 1623.1) includes updated acceptance criteria for IPR, OPR, and MS/MSD, and clarifications and revisions based on user questions and feedback about Method 1623 over the past 19 years.

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[1] Formerly known as the American Society for Testing and Materials (ASTM).

[2] NPDES permit regulations also specify that the approved method needs to be sufficiently sensitive. See 40 CFR 122.21.e.3.

B. Methods Incorporated by Reference

Currently, hundreds of methods and ATPs are incorporated by reference within 40 CFR part 136. In most cases, 40 CFR part 136 contains multiple approved methods for a single pollutant, and regulated entities often have a choice in selecting a method. The proposed rule contains revisions to VCSB methods that are currently incorporated by reference. Two VCSBs have made such revisions, Standard Methods and ASTM. The proposed VCSB methods are consistent with the requirements of the National Technology Transfer and Advancement Act (NTTAA), under which federal agencies use technical standards developed or adopted by the VCSBs if compliance would not be inconsistent with applicable law or otherwise impracticable (see Section V.I below). The proposed VCSB methods are available on their respective websites (*www.standardmethods.org/* and *www.astm.org*) to everyone at a cost determined by the VCSB, generally from $40 to $80. Both organizations also offer memberships or subscriptions that allow unlimited access to their methods. The cost of obtaining these methods is not a significant financial burden for a discharger or environmental laboratory, making the methods reasonably available. Finally, this proposal also includes USGS methods and vendor ATPs, all of which the EPA proposes to incorporate by reference. The ATPs and USGS methods are available free of charge on their respective websites (*flowinjection.com*, *mn-net.com*, *micrologylabs.com*, and *USGS.gov*), enabling the EPA to conclude that the USGS methods and ATPs incorporated by reference are reasonably available.

C. Changes to 40 CFR 136.3 To Include New Versions of Approved Standard Methods

The EPA is proposing to approve new versions of Standard Methods methods previously approved in 40 CFR part 136. The newer versions provide clarifications or make editorial corrections. As was the case with the previous methods update rule (82 FR 40836–40941, August 28, 2017), the EPA generally proposes to approve and include in 40 CFR part 136 only the most recent version of a method published by the Standard Methods Committee. The EPA is proposing to list only one version of the method with the year of publication designated by the last four digits in the method number (*e.g.,* Standard Methods Method 3111 B–2011). The date indicates the date of the specific revision to the method. This allows use of a specific method in any edition of the hard copy publication of *Standard Methods for the Examination of Water & Wastewater* (Standard Methods) that includes a method with the same method number and year of publication.

The proposed revisions to Standard Methods methods previously approved in 40 CFR part 136 will not affect the performance of the method. Below is a list of the Standard Methods methods the EPA is proposing to include in 40 CFR part 136. Each entry contains the proposed Standard Methods number and date, the parameter, and a brief description of the analytical method. The methods listed below are organized according to the table at 40 CFR part 136 in which they appear.

The EPA proposes to make the following changes to Tables IA and IH at 40 CFR part 136:

1. Standard Methods Method 9221 (B, E, F)–2014: Method 9221B–2014 Coliform (total); analyzes for total coliforms in non-potable waters using lauryl tryptose broth (LTB), all presumptive growth LTB tubes are confirmed in brilliant green lactose bile broth (BGLB). Method 9221E–2014 Coliform (fecal); analyzes all presumptive growth LTB tubes for fecal coliform using EC broth. Method 9221F–2014 *E. coli;* analyzes all presumptive growth LTB tubes for *E. coli* using EC–MUG. The number of positive tubes (BGLB, EC broth or EC–MUG) is used to determine the most probable number (MPN).

2. Standard Methods Method 9222 (B, D, I)–2015: Method 9222B–2015 Coliform (total); analyzes for total coliforms in non-potable waters by filtration through a 0.45-µm membrane filter and plated on mEndo or LES Endo agar. Method 9222D–2015 Coliform (fecal); analyzes for fecal coliforms in non-potable waters by filtration through a 0.45-µm membrane filter plated on mFC medium. Method 9222 I–2015 *E. coli;* membrane filtration (MF), analyzes presumptive positive filters from Method 9222B and 9222D using nutrient agar plates with MUG (NA–MUG) which are examined under a longwave UV lamp.

3. Standard Methods Method 9223B–2016, *E. coli,* multiple tube/multiple well. This method analyzes non-potable waters for *E. coli* using commercially available enzyme substrate media that is mixed with the sample and placed in multiple tubes or multiple well trays, incubated and examined under ambient light for Coliform (total) and under a longwave UV lamp for *E. coli.*

4. Standard Methods Method 9230 (B, C)–2013: Method 9230B–2013 Fecal Streptococci; analyzes non-potable waters for streptococci using azide dextrose broth (ADB), Presumptive positive ADB tubes are confirmed by streaking onto bile esculin azide agar (BEA). Method 9230C–2013 Enterococci; analyzes non-potable waters by filtration through a 0.45-µm membrane filter and plated on mE agar.

The EPA proposes to make the following changes to Table IB at 40 CFR part 136:

1. Standard Methods Methods: Method 2540B–2015, total solids; a sample aliquot is evaporated in a pre-weighed evaporating dish at 103–105 °C. Method 2540C–2015 filterable residue (total dissolved solids); a sample aliquot is filtered through a glass fiber filter and the filtrate is evaporated on a pre-weighed dish to constant weight at 180 °C. Method 2540D–2015 non-filterable residue (total suspended solids); a sample aliquot is filtered through a pre-weighed glass fiber filter which is then dried to constant weight at 103–105 °C. Method 2540E–2015 volatile residue (fixed and volatile solids); the residue obtained from the determination of total (Method 2540B), filterable (Method 2540C) or non-filterable residue (Method 2540D) is ignited at 550 °C in a muffle furnace. Method 2540E–2015 settleable residue (settleable solids); settleable matter is measured with an Imhoff cone either volumetrically or gravimetrically.

2. Standard Methods Method 4500–CN^- (B–G)–2016, cyanide: Cyanides are measured after preliminary treatment of samples to remove interferences (4500–CN^- B) and manual distillation with magnesium chloride ($MgCl_2$) (4500–CN^- C) followed by: Titration with silver nitrate (4500–CN^- D), spectrophotometric measurement after cyanide in the alkaline distillate is converted to CNCl (4500–CN^- E), potentiometric measurement using an ion selective electrode (4500–CN^- F), and cyanide amenable to chlorination (CATC) in which a portion of the sample is chlorinated at high pH and cyanide levels in the chlorinated sample are determined after manual distillation followed by titrimetric or spectrophotometric measurement. Amenable cyanide is calculated by the difference between the results for cyanide in the unchlorinated sample and the results for the chlorinated sample (4500–CN^- G).

3. Standard Methods Method 4500–NO_3^- D–2016, nitrate (as nitrogen), measured using an ion-selective electrode (ISE) that develops a potential across a thin, inert membrane holding in place a water-immiscible liquid ion exchanger.

4. Standard Methods Method 4500–NO_3^- (E, F, and H)–2016, nitrate-nitrite (as nitrogen): Nitrate is reduced to nitrite using a cadmium-copper column, followed by diazotization to form a colored azo dye, which is measured by colorimetry either manually (4500 NO_3^- E) or automated (4500 NO_3^- F); or by reduction of nitrate to nitrite using hydrazine followed by automated colorimetric measurement of nitrite after diazotization (4500 NO_3^- H).

5. Standard Methods Method 4500–NO_3^- (E and F)–2016, nitrite (as nitrogen), colorimetric: Bypasses the cadmium reduction step and measures nitrite after diazotization either by manual or automated colorimetric analysis.

6. Standard Methods Method 4500–O (B–F, and G)–2016, measurement of oxygen (dissolved), using the Winkler iodometric titration procedure with modifications to eliminate or minimize certain interferences if necessary based on sample type (4500–O B through F), or by use of polarographic or galvanic membrane electrodes (4500–O G).

7. Standard Methods Method 5210 B–2016, biochemical oxygen demand (BOD_5), dissolved oxygen depletion. The BOD_5 test is an indirect measurement of organic matter; it measures the change in DO concentration caused by microorganisms as they degrade organic matter in a sample held in a stoppered bottle incubated for 5 days in the dark at 20 °C.

8. Standard Methods Method 5310 (B, C)–2014, total organic carbon (TOC), combustion, heated persulfate or UV persulfate oxidation: Method 5310B–2014, high-temperature combustion; a sample aliquot is combusted, transported in a carrier gas stream and measured via a nondispersive infrared analyzer, or titrated coulometrically Method 5310C–2014, persulfate–ultraviolet or heated-persulfate oxidation method; persulfate oxidizes organic carbon, the produced CO_2 is then purged and measured by either nondispersive infrared (NDIR) analyzer, be coulometrically titrated, or separated from the liquid stream by a membrane that specifically allows CO_2 to pass into high-purity water where the change in the high-purity water's conductivity corresponds to the amount of CO_2 passing the membrane.

The EPA is also proposing one revision to a previously approved Standard Methods method for which the Standard Methods Committee has adopted updates. This modification includes minor procedural changes that do not affect the performance of the method.

The EPA proposes to make the following change to Table IA and Table IH at 40 CFR part 136:

1. The EPA proposes that the MPN method Standard Methods Method 9221 F.2–2014 be approved as an acceptable method for detecting thermotolerant coliforms and *E. coli* simultaneously. This method analyzes Coliform (fecal) and *E. coli* using EC broth with 4-methylumbelliferyl-β-D-glucuronide (EC–MUG) with inverted vials.

D. Changes to 40 CFR 136.3 To Include New Standard Methods Methods Based on Previously Approved Technologies

The EPA is proposing these changes based on the National Technology Transfer and Advancement Act of 1995 (NTTAA), Public Law 104–113, which states that federal agencies and departments shall use technical standards developed or adopted by the VCSBs if compliance would not be inconsistent with applicable law or otherwise impracticable. These methods submitted by the Standard Methods Committee are consistent with other methods already approved at 40 CFR part 136.

1. The EPA proposes to add Standard Methods Method 4500–CN^- N–2016 to Table IB for Cyanide, total. Cyanide is measured after preliminary treatment of samples and manual distillation with magnesium chloride ($MgCl_2$) followed by automated spectrophotometric measurement after conversion to CNCl. This method is similar to the currently approved EPA Method 335.4, USGS Method I–4302–85, and Lachat Method 10–204–00–1–X, and uses semi-automated spectrophotometric measurement of cyanide.

2. The EPA proposes to add Standard Methods Method 4500–NO_3^- I–2016 to Table IB for combined nitrate-nitrite, nitrite (bypass the reduction column) and nitrate by subtraction. Nitrate is reduced to nitrite using a cadmium-copper column followed by diazotization to form an azo dye which is measured by colorimetry. The cadmium reduction column may be by-passed for measurement of nitrite only. The value obtained for nitrite may be subtracted from the value obtained for combined nitrate-nitrite to calculate the concentration of nitrate. This method is similar to the currently approved EPA Method 353.2, Standard Methods Method 4500–NO_3^- F–2011, ASTM Method D3867–04 (A), and USGS Method I–2545–90, and uses automated cadmium reduction and spectrophotometric measurement of nitrite.

3. The EPA proposes to add Standard Methods Method 4500–NO_3^- J–2018 to Table IB for measurement of combined nitrate-nitrite, for measurement of nitrite when bypassing the enzymatic reduction step, and for measurement of nitrate by subtraction. Nitrate is reduced to nitrite by an enzymatic reaction. The nitrite is diazotized to yield an azo dye which is measured colorimetrically. The enzyme reduction step may be by-passed for measurement of nitrite singly. The value obtained for nitrite may be subtracted from the value obtained for combined nitrate-nitrite to calculate the concentration of nitrate. This method is similar to the currently approved NECi Method N07–0003, USGS Method I–2547–11, and USGS Method I–2548–11.

4. The EPA proposes to add Standard Methods Method 4500–O H–2016 to Table IB for dissolved oxygen. This method uses a luminescent-based sensor for measurement of dissolved oxygen. The method is similar to the currently approved Hach Method 10360, In-Situ Method 1002–8–2009, and ASTM Method D888–09 (C).

E. Changes to 40 CFR 136.3 To Include New Versions of Approved ASTM Methods

The EPA is proposing to approve new versions of ASTM methods previously approved in 40 CFR part 136 for the same reasons outlined in the first paragraph of Section IV.C above. These changes to currently approved ASTM methods in 40 CFR part 136 include minor clarifications and editorial changes, and in some instances, minor procedural changes. None of these proposed changes will affect the performance of the method. The following describes the changes to current ASTM methods that the EPA proposes to include in 40 CFR part 136. Each entry contains (in the following order): Proposed ASTM method number (the last two digits in the method number represent the year ASTM published), the parameter, a brief description of the analytical technique, and a brief description of any procedural changes in this revision from the last approved version of the method. The methods listed below are organized according to the table at 40 CFR part 136 in which they appear.

The EPA proposes the following changes to Table IB at 40 CFR part 136:

1. ASTM Method D511–14 (A, B), calcium and magnesium, titrimetric, (EDTA), AA direct aspiration: Method D511–14 A, titrimetric; the pH of the sample is adjusted to 10 (for calcium), then to 12–13 (for magnesium) and titrated with ethylenediamine tetraacetic acid (EDTA) to form complexes with calcium and

magnesium ions which react with an indicator to form a colored product. The volume of titrant used to affect the color change is proportional to the concentrations of calcium and magnesium in the sample. Method D511–14 B, AA direct aspiration; the sample is acidified and analyzed by atomic absorption. The concentrations of calcium and magnesium in the samples are proportional to the amount of light absorbed during the analysis, and are determined in comparison to a standard curve.

This version adds specifications for filter paper.

2. ASTM Method D512–12 chloride ion (A, B), titrimetric (mercuric nitrate), titration (silver nitrate): Method D512–12A, titrimetric mercuric nitrate; the sample is acidified and titrated with mercuric nitrate in the presence of a diphenylcarbazonebromophenol blue indicator. Method D512–12B, titrimetric silver nitrate; sample pH is adjusted to phenolphthalein endpoint and titrated with silver nitrate in the presence of potassium chromate. The volume of titrant used to affect the color change in either method is proportional to the concentration of chloride in the sample. This version corrects one term in the calculation of the chloride calculation.

3. ASTM Method D516–16, sulfate ion, turbidimetric. In this method, sulfate ions are converted to barium sulfate to form a suspension. The turbidity of the suspension is measured with a nephelometer, spectrophotometer, or photoelectric colorimeter, and compared to a standard curve to determine the sulfate concentration in the sample. This version adds specifications for filter paper.

4. ASTM Method D858–17 (A–C), manganese, atomic absorption (AA) direct aspiration, AA furnace. The sample is acid digested and analyzed by direct aspiration atomic absorption or graphite furnace atomic absorption. The concentration of manganese in the sample is proportional to the amount of light absorbed and is determined in comparison to a standard curve. There are no procedural changes.

5. ASTM Method D859–16, silica, colorimetric, manual. In this method, soluble silica in the sample is reacted with molybdate then reduced to form a blue complex in solution. The intensity of the blue complex is determined with a spectrophotometer or filter photometer and the concentration of silica is determined by comparison with a standard curve. There are no procedural changes.

6. ASTM Method D888–12 (A–C) dissolved oxygen, Winkler, electrode, luminescent-based sensor: Method D888–12A measures dissolved oxygen using the Winkler iodometric titration procedure. The volume of titrant used is proportional to the concentration of dissolved oxygen in the sample. Method D888–12B measures dissolved oxygen in the sample with an electrochemical probe that produces an electrical potential which is logarithmically proportional to the concentration of dissolved oxygen in the sample. Method D888–12C measures dissolved oxygen with a luminescence-based sensor probe that employs frequency domain lifetime-based luminescence quenching and signal processing. This version adds information on a two-point calibration and updated performance information from an interlaboratory study to D888–12C.

7. ASTM Method D1067–16, acidity or alkalinity, electrometric endpoint or phenolphthalein endpoint; electrometric or colorimetric titration to pH 4.5, manual. The acidity or alkalinity of the sample is determined by titration to a specific pH endpoint which is determined by colorimetry or with a pH electrode. The acidity or alkalinity is proportional to the volume of titrant required to affect the pH change. There are no procedural changes.

8. ASTM Method D1068–15 (A–C), iron, AA direct aspiration; AA furnace; colorimetric (Phenanthroline): The sample is acid digested and analyzed by either direct aspiration atomic absorption, graphite furnace atomic absorption, or colorimetry. The concentration of iron in the sample is proportional to the amount of light absorbed and is determined in comparison to a standard curve. This version adds specifications for filter paper.

9. ASTM Method D1126–17, hardness, titrimetric (EDTA). The pH of the sample is adjusted and an indicator is added forming a red color. The mixture is titrated until the color changes from red to blue. The volume of titrant used to affect the color change is proportional to the hardness in the sample. There are no procedural changes.

10. ASTM Method D1179–16 (A, B); fluoride ion, manual distillation, electrode, manual: Method D1179A, manual distillation; the sample is distilled as hydrofluorosilic acid and determined by ion-selective electrode. Method D1179B, electrode; the fluoride ion is determined potentiometrically with an ion-selective electrode in conjunction without sample distillation. There are no procedural changes.

11. ASTM Method D1246–16, bromide ion, electrode. The bromide in the sample is determined potentiometrically with an ion-selective electrode, either through comparison to a standard curve or through a direct readout on the instrument. There are no procedural changes.

12. ASTM Method D1252–06 (A, B) (Reapproved 2012), chemical oxygen demand, titrimetric, spectrophotometric. This is the 2012 reapproval of the 2006 ASTM method: Method D1252–06A, titrimetric; measures the loss of the hexavalent dichromate ion by reflux digestion followed by titration. The chemical oxygen demand in the sample is determined by comparison to a standard curve. Method D1252–06B, spectrophotometric; uses a spectrophotometer to measure the loss of the hexavalent dichromate ion at 420 nm or the increase in the trivalent chromium ion at 600 nm, after closed digestion and determines the chemical oxygen demand by comparison to a standard curve. There are no procedural changes.

13. ASTM Method D1253–14, residual chlorine, amperometric direct. The concentration of chlorine in the sample is determined by titration with phenylarsine oxide, using an amperometric probe that responds to chlorine to determine when the titration is complete. The chlorine concentration in the sample is proportional to the volume of titrant used. There are no procedural changes.

14. ASTM Method D1426–15 (A, B), ammonia nitrogen, Nesslerization, electrode: Method D1426A, Nesslerization; an aliquot is Nesslerized and the ammonia content determined colormetrically. Method D1426B, electrode; ammonia is potentiometric determined using a gas-permeable ion-selective electrode, either through comparison to a standard curve or through a direct readout on the instrument using. A lengthy section of QC requirements was added to the Nesslerization procedure (D1426A) that parallels the QC discussion that was already in the B procedure. Both procedures added information on use of commercially prepared standards and filter paper.

15. ASTM Method D1687–17 (A–C), chromium (total) and dissolved hexavalent chromium, colorimetric (diphenyl–carbazide); AA direct aspiration; AA furnace: Method D1687–17A, chromium (dissolved); measures dissolved hexavalent chromium by reacting it with diphenyl-carbohydrazide to produce a reddish-purple color that is measured with a

spectrophotometer or filter photometer. The concentration in the sample is proportional to the intensity of the color. Method D1687–17B, chromium (total); the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D1687–17C, chromium (total); the sample is acid digested and analyzed by graphite furnace atomic absorption. The concentration of total chromium in the sample is proportional to the amount of light absorbed during the analysis and is determined in comparison to a standard curve. The changes mirror those for the other metals methods. The QC frequencies for method blank, continuing calibration verification (CCV), continuing calibration blank (CCB), matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples.

16. ASTM Method D1688–17 (A–C), copper, AA direct aspiration, AA furnace. The sample is acid digested and analyzed by direct aspiration atomic absorption (D1688–17A and B) or graphite furnace atomic absorption (D1688–17B). The concentration of copper in the sample is proportional to the amount of light absorbed and is determined in comparison to a standard curve. The changes mirror those for the other metals methods. The proposed changes also clarify the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples.

17. ASTM Method D1691–17 (A, B), zinc, AA direct aspiration. Method D1691–17A; the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D1691–17B; the sample is processed by chelation-extraction and analyzed by atomic absorption. The concentration of zinc in the sample is proportional to the amount of light absorbed and is determined in comparison to a standard curve. The changes mirror those for the other metals methods. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples.

18. ASTM Method D1783–01 (A, B) (Reapproved 2012), phenols, manual distillation followed by manual colorimetric (4AAP). The sample is distilled, the distillate pH is adjusted to 10.0, and reacted with 4-aminoantipyrine to form a colored product. In Method D1783–01A, the colored product is extracted from the sample with chloroform and measured with a photometer at 460 nm. In Method D1783–01B, the colored product is measured without extraction, using a photometer at 510 nm. The concentration of phenolics is determined in comparison to a standard curve. There are no procedural changes.

19. ASTM Method D1886–14 (A–C), nickel AA direct aspiration, chelation extraction AA and AA furnace. Method D1886–14A, the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D1886–14B, the sample is acid digested and the nickel chelated and extracted. The extract is analyzed by direct aspiration atomic absorption. Method D1886–14C, the sample is acid digested and analyzed by graphite furnace atomic absorption. The concentration of nickel in the sample is proportional to the amount of light absorbed during the analysis and is determined in comparison to a standard curve. The changes mirror those for the other metals methods. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples.

20. ASTM Method D2036–09 (A, B) (Reapproved 2015), A, total cyanide, manual distillation followed by gas diffusion amperometry, titrimetric, spectrophotometric, ion chromatography, ion selective electrode, B, available (amenable) cyanide, manual distillation followed by titrimetric or spectrophotometric. The cyanide in the sample is distilled and trapped in a sodium hydroxide solution. Method D2036–09A, the cyanide is treated with strong acid and a catalyst during distillation and measured by titration, gas diffusion amperometry, spectrophotometry, ion-selective electrode, ion chromatography, or flow injection analysis. Method D2036–09B, cyanide amenable to chlorination is determined by comparing the results for one sample aliquot analyzed for total cyanide and a second aliquot that is treated with calcium hypochlorite prior to analysis by Method D2036–09A. There are no procedural changes.

21. ASTM Method D2972–15 (A–C), arsenic, colorimetric, AA gaseous hydride, AA furnace. The sample is digested with nitric and sulfuric acids. Method D2972–15A, arsenic is trapped in a solution of silver diethyldithiocarbamate in pyridine which produces a red-colored product that is analyzed photometrically by comparison to a standard curve. Method D2972–15B, arsenic in the digested sample is determined by hydride generation atomic absorption. Method D2972–15C, arsenic in the digested sample is determined by graphite furnace atomic absorption. The changes mirror those for the other metals methods. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples.

22. ASTM Method D3223–17, total mercury, cold vapor, manual. Mercury in the sample is converted to the mercuric ion which is reduced to elemental mercury, purged from the sample, and analyzed by cold vapor atomic absorption. The changes mirror those for the other metals methods, but this version changes the acceptance limit for the CCV from 10% to 15% and adds a requirement for a CCB. Given that the most comparable EPA procedure, Method 245.1, does not include a CCV requirement or an acceptance limit, the change of the acceptance limit from 10% to 15% in the revised ASTM method represents a requirement that is more stringent than that required in the EPA's procedure and therefore, the change to the ASTM method is not an impediment to its approval.

23. ASTM Method D3373–17, vanadium, AA furnace. The sample is digested with nitric acid and analyzed by graphite furnace atomic absorption. The concentration of vanadium in the sample is proportional to the amount of light absorbed during the graphite furnace atomic absorption analysis and is determined in comparison to a standard curve. The changes mirror those for the other metals methods. The proposed changes clarify the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples.

24. ASTM Method D3557–17 (A–D), cadmium, AA direct aspiration, voltammetry, AA furnace. Method D3557–17A, the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D3557–17B, the sample is acid digested, the digestate is chelated and extracted. The extract analyzed by direct aspiration atomic absorption. Method D3557–17C, the sample is acid digested and analyzed by differential pulse anodic stripping voltametry. Method D3557–17D, the sample is digested with nitric acid and analyzed by graphite furnace atomic absorption. The concentration of cadmium in the sample is determined in comparison to a standard curve. The changes mirror those for the other

metals methods. The proposed changes also clarify requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples, as opposed to 10 samples previously.

25. ASTM Method D3558–15 (A–C), cobalt, AA direct aspiration, chelation extraction AA, and AA furnace. Method D3558–15A, the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D3558–15B, the sample is acid digested, chelated and extracted. The extract is analyzed by direct aspiration atomic absorption. Method D3558–15C, the sample is acid digested and analyzed by graphite furnace atomic absorption. The concentration of cobalt in the sample is proportional to the amount of light absorbed during the analysis and is determined in comparison to a standard curve. The changes mirror those for the other metals methods. The proposed changes also clarify the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch of up to 20 samples, as opposed to 10 samples previously.

26. ASTM Method D3559–15 (A–D), lead, AA direct aspiration, voltammetry, AA furnace. Method D3559–15A, the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D3559–15B, the sample is acid digested, chelated and extracted. The extract is analyzed by direct aspiration atomic absorption. Method D3559–15C, the sample is acid digested and analyzed by differential pulse anodic stripping voltametry. Method D3559–15D, the sample is digested with nitric acid and analyzed by graphite furnace atomic absorption. The changes mirror those for the other metals methods. The proposed changes also clarify the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. It also adds a new section with the QC requirements to the direct AA procedure that was already present in the AA furnace portion of this procedure (D3559–15 [D]).

27. ASTM Method D3590–17 (A, B), total Kjeldahl nitrogen, manual digestion and distillation or gas diffusion; semi-automated block digester colorimetric (distillation not required). Method D3590–17A, the sample is chemically processed to covert nitrogenous compounds to ammonia, then distilled or subjected to a gas diffusion system which releases the ammonia for analysis by colorimetry, titrimetry, or potentiometry. Method D3590–17B, the digestion and distillation are accomplished by a semi-automated system and the resulting ammonia is determined by colorimetry of the salicylate/nitroprusside Berthelot reaction product. This version changes the acceptance limit for the CCV from 10% to 15% and adds a requirement for a CCB. Given that neither the approved Standard Methods method for measuring ammonia after the TKN digestion, nor the comparable EPA Method 350.1, include a CCV requirement or an acceptance limit, the change of the acceptance limit from 10% to 15% in the revised ASTM method represents a requirement that is more stringent than that required in other approved procedures and therefore is not an impediment to its approval.

28. ASTM Method D3645–15, beryllium (A, B), AA direct aspiration AA furnace. Method D3645–15A, the sample is acid digested and analyzed by direct aspiration atomic absorption. Method D3645–15B, the sample is digested with nitric acid and analyzed by graphite furnace atomic absorption. This version adds specifications for filter paper. The proposed changes also clarify the requirements for a three-point calibration by discussing it in the calibration section as well as the QC section of both procedures. It also adds a new section with the QC requirements to the direct aspiration AA procedure that was already present in the AA furnace portion of this procedure (D3645–15B).

29. ASTM Method D3859–15 (A, B), selenium, AA gaseous hydride, AA furnace. In Method D3859–15A, the selenium in the sample is converted to gaseous selenium hydride, which is then analyzed by flame atomic absorption. Method D3859–15B, the selenium in the sample is converted to gaseous selenium hydride and analyzed by graphite furnace atomic absorption. The changes to the gaseous hydride portion of the method clarify the requirement for a 6-point calibration curve by discussing it in the calibration section as well as the QC section. The version adds an updated discussion of block digesters. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now based on a laboratory-defined batch, as opposed to an otherwise undefined ‘‘batch.’’ The GFAA portion contains similar editorial and technical changes. Technical changes also include specifications for filter paper. The calibration requirement for three standards has been clarified by discussing it in the calibration section as well as the QC section.

30. ASTM Method D3867–16 (A, B) nitrate-nitrite, nitrite and nitrate; automated cadmium reduction, manual cadmium reduction, bypass cadmium reduction and subtraction. The combination of nitrate and nitrite in the sample is determined by reducing the nitrate to nitrite using a cadmium-copper column, diazotizing and analyzing in either a manual or automated spectrophotometric system. A second aliquot of the sample can be analyzed without use of the cadmium reduction column to determine the concentration of nitrate by difference. The changes add more detailed QC requirements, including specifically calling out the laboratory control sample (LCS), method blank, and matrix spike analyses. The 2016 version adds specifications for filter paper. It also changes the LCS frequency from 10% of samples to once per batch (up to 20) and sets the CCB and CCV frequencies at 10%.

31. ASTM Method D4190–15, dissolved elements and total recoverable elements, direct current plasma. The concentrations of various metal elements are determined by acidifying an aliquot of the sample and analyzing it by direct current plasma spectrometry, monitoring a specific wavelength of light for each element. There is one change that adds a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.

32. ASTM Method D4282–15, free cyanide, manual micro-diffusion and colorimetry. The sample is treated and allow for free cyanide diffuse into a sodium hydroxide solution. An aliquot of that solution is treated to form a colored product that is measured with a spectrophotometer at 580 nm. There are no procedural changes.

33. ASTM Method D4327–17, inorganic anions (fluoride, bromide, chloride, nitrite, nitrate, orthophosphate, and sulfate), ion chromatography. An aliquot of the sample in injected into an ion chromatograph equipped with an anion exchange column and a conductivity detector. The anions are identified based on their retention times and concentrations are determined by comparison to a standard curve. Changes include updating the equipment and reagent descriptions to

reflect more modern instrumentation, such as the use of hydroxide eluents and eluent regeneration systems.

34. ASTM Method D4382–18, barium, AA furnace. The sample is digested with nitric acid and analyzed by graphite furnace atomic absorption. The only procedural change is to the description of the hot block digester equipment. The new version specifies the capability to heat samples between 65 and 95 degrees C, instead of ''approximately 95 degrees C.'' That change recognizes the operational characteristics of hot block digesters that will experience a temperature drop below 95 degrees when samples are added. This should not adversely affect use of this method for barium.

35. ASTM Method D4658–15, sulfide ion, ion selective electrode. The sample is treated with a sulfide antioxidant buffer to create a highly alkaline solution. Sulfide in the sample is measured potentiometrically with an ion-selective electrode. There are no procedural changes.

36. ASTM Method D4839–03 (Reapproved 2017), total organic carbon; heated persulfate or UV persulfate oxidation. The sample is sparged with an inert gas to remove dissolved inorganic carbon and then treated with persulfate and either heat or UV radiation to convert organic carbon to carbon dioxide. The carbon dioxide is measured with an infra-red detector. There are no procedural changes.

37. ASTM Method D5257–17, dissolved hexavalent chromium, ion chromatography. The sample is filtered and buffered and an aliquot injected into an ion chromatograph that separates hexavalent chromium from other ions. The eluent from the chromatograph is treated with an acidic solution of diphenylcarbohydrazide to form a violet-colored product that is measured with a photometric detector at 530 nm. The changes add a few additional warnings or recommendations.

38. ASTM Method D5673–16, dissolved elements and total-recoverable elements, ICP/MS. The sample is acid digested and analyzed by inductively coupled plasma/mass spectrometry. Gold was added to the list of target analytes. Some of the changes address the analysis of gold.

39. ASTM Method D6508–15, inorganic anions (fluoride, bromide, chloride, nitrite, nitrate, orthophosphate, and sulfate), capillary ion electrophoresis with indirect UV detection. An aliquot of the sample in injected into a capillary ion electrophoresis instrument where the anions are separated in an applied electric field through a fused silica capillary. The analytes are detected by a UV detector and their concentrations are determined by comparison to a standard curve. There are no procedural changes.

40. ASTM Method D6888–16, available cyanide, flow injection and ligand exchange, followed by gas diffusion amperometry. An aliquot of the sample is introduced into a flow injection analysis instrument, where available cyanide is acidified to form hydrogen cyanide which diffuses through a hydrophobic gas diffusion membrane into an alkaline solution and is detected amperometrically with a silver electrode. This version adds a new mixed ligand exchange reagent, but also retains the original two ligand reagents that had to be mixed together during the testing.

41. ASTM Method D6919–17, inorganic alkali and alkaline earth cations and ammonium (ammonium, calcium magnesium, potassium and sodium), ion chromatography. An aliquot of the sample in injected into an ion chromatograph equipped with a cation exchange column and a conductivity detector. The cations are identified based on their retention times and concentrations are determined by comparison to a standard curve. There are no procedural changes.

42. ASTM Method D7237–15 (A), free cyanide, flow injection, followed by gas diffusion amperometry. An aliquot of the sample is introduced into a flow injection analysis instrument, where it mixes with a phosphate buffer to release hydrogen cyanide which diffuses through a hydrophobic gas diffusion membrane into an alkaline solution and is detected amperometrically with a silver electrode. There are a few additions and changes to the newer version of note. The statement of the applicable range of the method in Section 1.4 has been changed at the low end, from 2 to 500 µg/L to 5 to 500 µg/L. New information about interferences from floatation reagents has been added to Section 6.3. New materials in Section 8 discuss alternative reagents or concentrations.

43. ASTM Method D7284–13 (Reapproved 2017), total cyanide, manual distillation with $MgCl_2$ followed by flow injection, gas diffusion amperometry. The sample is distilled with acid and a magnesium chloride catalyst to release cyanide to a sodium hydroxide solution. An aliquot of the sodium hydroxide solution is introduced into a flow injection analysis instrument, where it is acidified and the hydrogen cyanide diffuses through a hydrophobic gas diffusion membrane into an alkaline solution and is detected amperometrically with a silver electrode. There are no procedural changes.

44. ASTM Method D7511–12 (Reapproved 2017), total cyanide, segmented flow injection, in-line ultraviolet digestion, followed by gas diffusion amperometry. The sample is introduced into a segmented flow injection analysis instrument, where UV light releases cyanide from cyanide complexes. The sample is then acidified in the instrument and the produced cyanide gas is detected amperometrically with a silver electrode. There are no procedural changes.

45. ASTM Method D7573–09 (Reapproved 2017), total organic carbon, combustion. The sample is sparged with an inert gas to remove dissolved inorganic carbon, acidified, and then combusted at high temperature convert organic carbon to carbon dioxide. The carbon dioxide is measured with an infra-red detector. There are no procedural changes.

The EPA proposes the following changes to Table IC at 40 CFR part 136:

1. ASTM Method D7065–17, nonylphenol, bisphenol A, p-tert-octylphenol, nonylphenol monoethoxylate, nonylphenol diethoxylate, gas chromatography/mass spectrometry (GC/MS). The sample is extracted with methylene chloride and the extract is injected into a gas chromatograph-mass spectrometer. The target analytes are identified by retention time and mass spectra and quantified using internal standards and a calibration curve. There are a large number of editorial and structural changes in the document. A new QC section has been added.

F. Changes to 40 CFR 136.3 To Include a New ASTM Method Based on Previously Approved Technologies

The EPA is proposing these changes based on the National Technology Transfer and Advancement Act of 1995 (NTTAA), Public Law 104–113, which states that federal agencies and departments shall use technical standards developed or adopted by the VCSBs if compliance would not be inconsistent with applicable law or otherwise impracticable. This method submitted by ASTM is consistent with other already approved methods.

1. The EPA proposes to add ASTM Method D7781–14 to Table IB for nitrate-nitrite, nitrite (bypass the enzymatic reduction step) and nitrate by subtraction. Nitrate is reduced to nitrite by an enzymatic reaction. The nitrite is diazotized to yield an azo dye which is

measured colorimetrically. The enzyme reduction step may be by-passed for measurement of nitrite singly. The value obtained for nitrite may be subtracted from the value obtained for combined nitrate-nitrite to calculate the concentration of nitrate. This method is similar to the currently approved NECi Method N07–0003, USGS Method I–2547–11, and USGS Method I–2548–11.

G. Changes to 40 CFR 136.3 To Include New United States Geological Survey (USGS) Inorganic Methods Based on Previously Approved Technologies

1. The EPA proposes to add USGS Method I–2057–85 titled ''Anions, ion-exchange chromatographic, automated,'' to Table IB for bromide. Method I–2057–85 is an ion chromatography method that lists several target analytes: bromide, chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate. These are the same target analytes found in EPA Methods 300.0 (Part A) and 300.1 (Part A). Both EPA methods are approved in 40 CFR part 136 for the target analytes listed in the methods. USGS Method I–2057–85 is similar to EPA Method 300.0, in that it uses ion chromatography with a sodium bicarbonate/sodium carbonate eluent and has the same target analyte list. The two methods specify different columns and eluent concentrations, but rely on essentially the same underlying chemistry and determinative technique as other ion chromatography methods approved at 40 CFR part 136 for measurement of bromide. That is, the sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

2. The EPA proposes to add USGS Method I–2522–90 titled ''Nitrogen, ammonia, colorimetry, salicylate-hypochlorite, automated-segmented flow'' to Table IB for ammonia. USGS Method I–2522–90 uses the same underlying chemistry and determinative technique as other methods approved at 40 CFR part 136 for measurement of ammonia. The method is similar to other approved methods, such as EPA Method 350.1, Standard Methods Method 4500–NH3 G, and USGS Method I–4523–85, which rely on the Berthelot reaction. USGS Method I–2522–90 uses a modified version of the Berthelot reaction in which salicylate and hypochlorite react with ammonia in the presence of ferricyanide ions to form the salicylic analog of indophenol blue dye. The resulting color is directly proportional to the concentration of ammonia present and is measured using automated spectrophotometry. This is a well-documented modification to the Berthelot reaction used in EPA Method 351 and is specifically allowed in Table IB.

3. The EPA proposes to add USGS Method I–2540–90 titled ''Nitrogen, nitrite, colorimetry, diazotization, automated-segmented flow'' to Table IB for nitrite. USGS Method I–2540–90 employs the same underlying chemistry and determinative technique as other methods approved at 40 CFR part 136 for measurement of nitrite. The method is similar to other methods approved at 40 CFR part 136 for measurement of nitrite, including USGS Method I–4540–85, which uses an automated-segmented flow analyzer (Technicon AA II). Method I–2540–90, nitrite reacts with sulfanilamide under acidic conditions to form a diazo compound which is coupled with N–1-naphthylethylenediamine dihydrochloride to form a red compound, the absorbance of which is measured using an automated-segmented flow, spectrophotometry.

4. The EPA proposes to add USGS Method I–2601–90 titled ''Phosphorus, orthophosphate, colorimetry, phosphomolybdate, automated-segmented flow'' to Table IB for orthophosphate. USGS Method I–2601–90 employs the same underlying chemistry and determinative technique as other methods approved in 40 CFR part 136 for measurement of orthophosphate. Orthophosphate reacts with ammonium molybdate in acidic solution to form phosphomolybdic acid, which upon reduction with ascorbic acid produces an intensely blue complex the absorbance of which is measured using automated spectrophotometry. Antimony potassium tartrate is added to increase the rate of reduction. The method is similar to other approved methods, such as USGS Method I–4601–85 which uses an automated-segmented flow analyzer (Technicon AA II). The submitted USGS Method I–2601–90 also uses an automated-segmented flow analyzer (Alpkem rapid flow analyzer). It should be noted that the approved USGS Method I–4601–85 has two parameter codes listed:

a. Phosphorus, orthophosphate, dissolved, I–2601–85 (mg/L as P);

b. Phosphorus, orthophosphate, total, I–4601–85 (mg/L as P).

Although USGS Method I–4601–85 is listed in Table IB, samples to be used for measurement of orthophosphate are to be filtered upon collection per Table II. Therefore, the correct parameter code listed for the method should have been I–2601–85. I–2601–90 is just an updated version of that method (parameter code). In Section 3—Interferences, USGS Method I–2601–85 states: ''Because as phosphorus is easily adsorbed on sediment, the orthophosphate recovered from the supernatant solution above a water-suspended sediment after some time has elapsed may be less than the orthophosphate that would have been determined in the filtrate from a sample filtered at the time of collection. The amount recovered may also depend on the type of sediment (clay, sand, etc.).''

5. The EPA proposes USGS Method I–4472–97 titled ''Metals, Acid Digestion, Whole-Water Recoverable, inductively coupled plasma-mass spectrometry'' to be added to Table IB for certain metals by ICP/MS. USGS Method I–4472–97 is an ICP/MS method that was previously listed under the same method number as the USGS ICP/AES Method I–4471–97 and was split out and assigned a unique method number by USGS in 2003. The EPA proposes to add this to Table IB on the line for ICP/MS and replace USGS Method I–4471–97 as an approved method for measurement of the following 16 elements: aluminum, antimony, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium and zinc. USGS Method I–4472–97 relies on the same underlying chemistry and determinative technique as other ICP/MS methods approved at 40 CFR part 136 for measurement of the same 16 elements (*e.g.,* EPA Method 200.8 and Standard Methods Method 3125 B) where analytes in the sample are solubilized by gentle refluxing with acids and then measured using inductively coupled plasma-mass spectrometry.

H. Changes to 40 CFR 136.3 To Include New United States Geological Survey (USGS) Organic Methods Based on Previously Approved Technologies

1. The EPA proposes to add USGS Method O–4127–96 titled ''Determination of 86 Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry, Including Detections Less Than Reporting Limits'' to Table IC for certain organic compounds. USGS Method O–4127–96 relies on the same underlying chemistry and determinative technique as other methods approved at 40 CFR part 136 for measurement of the analytes for which the method is being proposed. Volatile organic compounds are extracted by purging with Helium, collecting onto a sorbent trap, thermally desorbed, separated by a gas chromatographic capillary column, and finally determined by a full-scan

quadrupole mass spectrometer. Compound identification is confirmed by the gas chromatographic retention time and by the resultant mass spectrum, typically identified by three unique ions.

2. The EPA Proposes to add USGS Method O–4436–16 titled ''Determination of Heat Purgeable and Ambient Purgeable Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry'' to Table IC for certain organic compounds. USGS Method O–4436–16 relies on the same underlying chemistry and determinative technique as other methods approved at 40 CFR part 136 for measurement of the analytes for which the method is being proposed. Volatile organic compounds are extracted from a water sample and compounds are trapped in a tube containing a suitable sorbent materials and then thermally desorbed into a capillary gas chromatographic column interfaced to a mass spectrometer system. Selected compounds are identified by using strict qualification criteria, which include analyzing standard reference materials and comparing retention times and relative ratios of the mass spectra. Compounds are quantitated using internal standard procedures.

I. Changes to 40 CFR 136.3 To Include Alternate Test Procedures (ATPs)

To promote method innovation, the EPA maintains a program that allows method developers to apply for EPA review and potential approval of an alternative method to an existing approved method. This ATP program is described for CWA applications at 40 CFR 136.4 and 136.5. The EPA is proposing three ATPs for nationwide use. Based on the EPA's review, the performance of these ATPs is equally effective as other methods already approved for measurement. The ATP applicants supplied EPA with study reports that contain the data from their validation studies. These study reports and the letters documenting EPA's review are contained as supporting documents within the docket for this proposed rule. These proposed new methods include: FIAlab Method 100, ''Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Fluorescence Detector Analysis,'' MACHEREY-NAGEL GmbH and Co. Method 036/038 NANOCOLOR® COD LR/HR, ''Spectrophotometric Measurement of Chemical Oxygen Demand in Water and Wastewater,'' and Micrology Laboratories, LLC. KwikCount™ EC Medium *Escherichia coli* (*E. coli*) enzyme substrate test, ''Rapid Detection of *E. coli* in Beach Water by KwikCount™ EC Membrane Filtration.'' Descriptions of these new methods proposed for approval are as follows:

1. FIAlab Instruments, Inc. Method 100, ''Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Fluorescence Detector Analysis,'' dated April 4, 2018 (FIAlab Instruments, Inc. 2018a). FIAlab Method 100 uses automated flow injection analysis with gas diffusion and fluorescence detector analysis to determine concentrations of ammonia in wastewater, ambient water, and Kjeldahl digestates. The method involves the following steps:

- The sample is introduced to the analyzer where it is made alkaline with sodium hydroxide;
- Ammonia is separated from the sample matrix by passage through a gas diffusion cell;
- After separation in the gas diffusion cell, ammonia is reacted with o-phthalaldehyde to form a fluorescent compound;
- The reaction product is detected by a fluorimeter and the response is directly proportional to the concentration of ammonia in the sample.

FIAlab Method 100 can be obtained from FIAlab Instruments, Inc., 334 2151 N Northlake Way, Seattle, WA 98103. Telephone: 425–376–0450.

2. MACHEREY-NAGEL GmbH and Co. Method 036/038 NANOCOLOR® COD LR/HR, ''Spectrophotometric Measurement of Chemical Oxygen Demand in Water and Wastewater,'' Revision 1.5, dated, May 2018 (MACHEREY-NAGEL GmbH and Co. 2018a). MACHEREY-NAGEL Method 036/038 NANOCOLOR® COD LR/HR is a manual method that uses spectrophotometry to measure chemical oxygen demand in wastewater. The method involves the following steps:

- Chemical Oxygen Demand (COD) is defined as the mg of oxygen (O_2) consumed per liter of sample following dichromate and sulfuric acid digestion;
- A sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to the green chromic ion (Cr^{3+});
- When the COD LR 150 test kit is used, the amount of Cr^{6+} remaining after digestion is determined;
- When the COD HR 1500 test kit is used, the amount of Cr^{3+} produced is determined.

MACHEREY-NAGEL GmbH and Co. Method 036/038 NANOCOLOR® COD LR/HR, can be obtained from MACHEREY-NAGEL GmbH and Co., 2850 Emrick Blvd., Bethlehem, PA 18020. Telephone: 888–321–6224.

3. Micrology Laboratories LLC. KwikCount™ EC Medium *E. coli* enzyme substrate test, ''Rapid Detection of *E. coli* in Beach Water by KwikCount™ EC Membrane Filtration'' uses a membrane filtration procedure for rapid detection and enumeration of *E. coli* in ambient water. The method involves the following steps:

- A water sample is filtered through a 0.45-µm pore size, 47-mm diameter membrane filter;
- The filter is then placed into a 50-mm plate containing an absorbent pad containing KwikCount™ EC broth;
- Plates are incubated at 41 ± 0.5 °C for 8–10 hr. The plates are then viewed in the dark using a long-wave UV light and fluorescent colonies are counted as *E. coli.*

The KwikCount™ EC Medium *E. coli* enzyme substrate test can be obtained from Micrology Laboratories, LLC, 1303 Eisenhower Drive, Goshen, IN 46526. Telephone: 574–533–3351.

J. Changes to 40 CFR 136.3, Tables IA, IB, and IH

The EPA is proposing the following changes to 40 CFR 136.3, Tables IA and IH:

1. Table IA: Moving Colilert-18 from Parameter #1 Coliform (fecal), number per 100 mL or number per gram dry weight, to Parameter #2 Coliform (fecal), (number per 100 mL), to eliminate confusion as to whether it is approved for sewage sludge in addition to wastewater.

2. Table IA: Adding *E. coli,* number per 100 mL—MF, two-step, Standard Methods Method 9222 B/9222 I, to the table along with footnote 31 ''Subject coliform positive samples determined by 9222 B–2015 or other membrane filter procedure to 9222 I–2015 using NA–MUG media.'' The method was inadvertently omitted from Table IA when Table IA was split into two tables (IA and IH) in an earlier rulemaking; the addition corrects that error.

3. Table IA: Revising Parameter #2 Coliform (fecal), deleting ''in presence of chlorine,'' number per 100 mL. The phrase ''in the presence of chlorine'' caused confusion because the methods cited were the same for the analyte/matrix combination that did not state ''in the presence of chlorine.'' The approved methods did not change.

4. Table IA: Deleting Parameter #4 Coliform (total) in presence of chlorine, number per 100 mL. Except for ''MF with enrichment,'' all the methods were duplicative (*e.g.,* Parameters #3 and #4).

No approved methods for coliform (total) were removed from Table IA.

5. Table IH: Deleting Parameters #2 Coliform (fecal) in presence of chlorine, number per 100 mL and #4 Coliform (total) in presence of chlorine, number per 100 mL. Except for ''MF with enrichment'' for coliform (total), all the methods were duplicative (*e.g.,* Parameters #1 and #2). In addition to the methods being duplicative, Table IH is for ambient water which would not be expected to contain chlorine. No approved methods for coliform (fecal) or coliform (total) were removed from Table IH. The remaining parameters are renumbered.

6. Tables IA and IH: Revising footnote 13 to Table IA and footnote 12 to Table IH as follows ''These tests are collectively known as defined enzyme substrate tests.'' The remaining text, ''where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by *E. coli*'' has been deleted because the example has caused some confusion to stakeholders.

7. Tables IA and IH: Adding Quanti-Tray®/2000 as an option to footnotes 13 (IH), 15 (IH), 16 (IA) and 18 (IA). The addition of Quanti-Tray®/2000 is to address matrices with high bacterial concentrations and to ensure Tables IA and IH are accurate and consistent.

8. Tables IA and IH: Adding footnote 30 to Table IA and footnote 27 to Table IH to specify a verification procedure. The footnotes contain the following language: ''On a monthly basis, at least ten sheen colonies from positive samples must be verified using Lauryl Tryptose Broth and brilliant green lactose bile broth, followed by count adjustment based on these results; and representative non-sheen colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.'' Adding the footnotes address the change in Standard Methods Method 9222 B–2015 that stated that five typical and five atypical colonies should be verified per membrane, which could be burdensome to laboratories analyzing samples other than drinking water. In most cases, analysis of ambient waters and wastewaters could result in multiple plates per sample with typical and atypical colonies, whereas drinking water analyses would seldom result in any typical or atypical colonies. In addition, the language in footnotes 29 (IA) and 26 (IH), was revised as follows ''the medium'' was replaced with ''positive samples'' for clarity and consistency.

9. Tables IA and IH: Adding footnote 32 to Table IA and footnote 30 to Table IH. The footnotes contain the following language ''Verification of colonies by incubation of BHI agar at 10 ± 0.5 °C for 48 ± 3 h is optional.'' As per the Errata to the 23rd Edition of Standard Methods for the Examination of Water & Wastewater, ''Growth on a BHI agar plate incubated at 10 ± 0.5 °C for 48 ± 3 h is further verification that the colony belongs to the genus Enterococcus.''

10. Table IH: Deleting ''or number per gram dry weight'' from Parameter #1. Table IH is specifically for ambient waters, which does not require reporting results on a per gram dry weight basis.

11. Table IH: Adding the Alternate Test Procedure KwikCount™ EC for *E. coli,* number per 100 mL under ''Other.''

12. Table IH: Adding EPA Method 1623.1 for Parameters 6 and 7. EPA Method 1623.1 includes updated acceptance criteria for IPR, OPR, and MS/MSD, and clarifications and revisions based on the use of EPA Method 1623 and technical support questions over the past 19 years. Both methods 1623 and 1623.1 will be listed as approved in the MUR because use of either method is acceptable.

13. Table IH: Deleting footnote 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.'' Table IH is specifically for ambient waters, so the footnote is not applicable. The remaining footnotes are renumbered accordingly.

14. Table IH: Revising footnote 20, to reference only EPA Method 1604. The literature reference was deleted from the footnote because it resulted in confusion as to whether EPA Method 1604 provided all the necessary information required by stakeholders to conduct analyses of ambient waters under the CWA.

K. Changes to Table II at 40 CFR 136.3(e) to Required Containers, Preservation Techniques, and Holding Times

The EPA is proposing to update footnote 6 to the preservation and holding time requirements for cyanide to cite the latest version of ASTM method D7365–09a that was reapproved in 2015. The recommended sampling and preservation procedures in the ASTM method have not changed since 2009, but the change to footnote 6 will simplify identification of the current method that is available from ASTM International.

The EPA is proposing to add footnote 9 to the preservation and holding time requirements to the purgeable halocarbons entry. This will allow the flexibility to collect a single sample with no acidification to be used for analysis of both purgeable halocarbons and purgeable aromatic hydrocarbons within seven days of collection, or to collect a single sample with acidification to be used for analysis of both purgeable halocarbons (except 2–CEVE) and purgeable aromatic hydrocarbons within the 14-day maximum holding time specified in Table II for both classes of compounds. The added flexibility is consistent with historical requirements for preservation in 40 CFR part 136 and holding time requirements in other EPA program methods, such as the SW–846 methods in the Office of Land and Emergency Management. This is part of the EPA's ongoing effort to harmonize methods between EPA programs, as requested by the Environmental Laboratory Advisory Board (ELAB).

Footnote 9 to Table II states: ''If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.''

L. Changes to 40 CFR 136.6 Method Modifications and Analytical Requirements

In response to requests from ELAB and the Independent Laboratories Institute (ILI), the EPA is proposing to add a new paragraph (b)(4)(xviii) to 40 CFR 136.6 that explicitly allows the use of closed-vessel microwave digestion as a modification to the approved metals digestion procedure that does not require prior approval. Microwave digestion has the same fundamental chemistry as a hot plate digestion, both the microwave and hot plate serve the same function as heat sources.

V. Statutory and Executive Order Reviews

A. Executive Order 12866: Regulatory Planning and Review and Review and Executive Order 13563: Improving Regulation and Regulatory Review

This rule is not a significant regulatory action and was therefore not submitted to the Office of Management and Budget (OMB) for interagency review under this E.O.

B. Paperwork Reduction Act

This action does not impose an information collection burden under the Paperwork Reduction Act. This rule does not impose any information collection, reporting, or recordkeeping requirements. This proposal would merely add or revise CWA test procedures.

C. Regulatory Flexibility Act

I certify that this action would not have a significant economic impact on a substantial number of small entities under the Regulatory Flexibility Act. This action will not impose any requirements on small entities. This action would approve new and revised versions of CWA testing procedures. Generally, these changes would have a positive impact on small entities by increasing method flexibility, thereby allowing entities to reduce costs by choosing more cost-effective methods. In general, the EPA expects the proposed revisions would lead to few, if any, increased costs. As explained previously, most of the proposed changes clarify or improve the instructions in the method, update the technology used in the method, improve the QC instructions, make editorial corrections, or reflect the most recent approval year of an already approved method. In some cases, the proposal would add alternatives to currently approved methods for a particular analyte (*e.g.,* Method N07–0003 for Nitrate Reductase Nitrate-Nitrogen Analysis). Because these methods would be alternatives rather than requirements, there are no direct costs associated with this proposal. The EPA proposes methods that would be incorporated by reference. If a permittee elected to use these methods, they could incur a small cost associated with obtaining these methods from the listed sources. See Section IV.B.

D. Unfunded Mandates Reform Act

This action does not contain any unfunded mandate as described in the Unfunded Mandates Reform Act, 2 U.S.C. 1531–1538, and does not significantly or uniquely affect small governments. The action imposes no enforceable duty on any state, local or tribal governments or the private sector.

E. Executive Order 13132: Federalism

This proposed rule does not have federalism implications. It will not have substantial direct effects on the states, on the relationship between the national government and the states, or on the distribution of power and responsibilities among the various levels of government.

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

This proposed rule does not have tribal implications as specified in Executive Order 13175. This rule would merely approve new and revised versions of test procedures. The EPA does not expect the proposal would lead to any costs to any tribal governments, and if incurred, projects they would be minimal. Thus, Executive Order 13175 does not apply to this action.

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

The EPA interprets Executive Order 13045 as applying only to those regulatory actions that concern environmental health or safety risks that the EPA has reason to believe may disproportionately affect children, per the definition of ‘‘covered regulatory action’’ in section 2–202 of the Executive Order. This action is not subject to Executive Order 13045 because it does not concern an environmental health risk or safety risk.

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

This action is not subject to Executive Order 13211 because it is not a significant regulatory action under Executive Order 12866.

I. National Technology Transfer and Advancement Act of 1995

This action involves technical standards. The EPA proposes to approve the use of technical standards developed and recommended by the Standard Methods Committee and ASTM International for use in compliance monitoring where the EPA determined that those standards meet the needs of CWA programs. As described above, this proposal is consistent with the NTTAA.

J. Executive Order 12898: Federal Actions To Address Environmental Justice in Minority Populations and Low-Income Populations

The EPA believes the human health or environmental risk addressed by this action will not have potential disproportionately high and adverse human health or environmental effects on minority, low-income or indigenous populations.

List of Subjects in 40 CFR Part 136

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

Dated: June 11, 2019.

Andrew R. Wheeler,

Administrator.

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

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

■ 1. The authority citation for part 136 continues to read as follows:

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

■ 2. Amend § 136.3 by:

■ a. In paragraph (a), seventh sentence, removing the word ‘‘year’’ and adding in its place the word ‘‘date’’ in its place, and removing from the last sentence the text ‘‘(paragraph (c) of this section, in § 136.5(a) through (d) or 40 CFR 401.13)’’ and adding in its place the text ‘‘paragraph (c) of this section, § 136.5(a) through (d) or 40 CFR 401.13,’’ respectively;

■ b. Revising tables IA, IB, IC, and IH;

■ c. Revising paragraph (b) by:

■ i. Revising the introductory text; paragraph (b)(8) introductory text, and paragraphs (b)(8)(ix) through (b)(xv);

■ ii. Adding paragraph (b)(8)(xvi);

■ iii. Revising paragraphs (b)(10)(xiv), (b)(10)(xxxix), (b)(10)(xliv), (b)(10)(xlvi), (b)(10)(lii), (b)(10)(liv), (b)(10)(lxvii), (b)(10)(lxviii), (b)(10)(lxix), (b)(10)(lxx), b)(15)(v), (b)(15)(vi), (b)(15)(viii) through (xiii), (b)(15)(xv) through (xix), (b)(15)(xxi) through (xxvi), (b)(15)(xxxi), (b)(15)(xxxiv) and (xxxv), (b)(15)(xxxvii), (b)(15)(xxxix) through (xliii), (b)(15)(xlv) through (l), (b)(15)(lii), (b)(15)(liv) and (b)(15)(lv), (b)(15)(lviii), (b)(15)(lxi) through (lxvi), and (b)(15)(lxviii) through (lxix); and

■ iv. Adding paragraph (b)(15)(lxx);

■ v. Redesignating paragraphs (b)(25) through (b)(36) as paragraphs (b)(28) through (b)(39);

■ vi. Redesignating paragraphs (b)(19) through (24) as paragraphs (b)(20) through (25);

■ vii. Adding new paragraphs (b)(19), (26), and (27); and

■ viii. Revising the newly redesignated paragraphs (b)(38)(ii) through (xxi);

■ ix. Adding paragraphs (b)(38)(xxii) and (xxiii); and

■ c. Revising paragraph (e) Table II.

The revisions and additions read as follows:

§ 136.3 Identification of test procedures.

* * * * *

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

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] 1680,[11,15] 1681,[11,20].	9221 E–2014.		
	Membrane filter (MF)[2,5], single step	p. 124[3]	9222 D–2015[29]	B–0050–85[4].	
2. Coliform (fecal), number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 132[3]	9221 E–2014; 9221 F.2–2014[33].		
	Multiple tube/multiple well, or				Colilert-18®.[13,18,28]
	MF[2,5], single step[5]	p. 124[3]	9222 D–2015[29].		
3. Coliform (total), number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B–2014.		
	MF[2,5], single step or two step	p. 108[3]	9222 B–2015[30]	B–0025–85[4].	
	MF[2,5], with enrichment	p. 111[3]	9222 (B+B.4e) – 2015[30].		
4. *E. coli,* number per 100 mL	MPN[6,8,16] multiple tube, or		9221 B.3–2014/9221 F–2014[12,14,33].		
	multiple tube/multiple well, or		9223 B–2016[13]	991.15[10]	Colilert®.[13,18] Colilert-18®.[13,17,18]
	MF[2,5,6,7,8], two step, or		9222 B–2015/9222 I–2015[31].		
	Single step	1603[21]			m-ColiBlue24®.[19]
5. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 139[3]	9230 B–2013.		
	MF[2], or	p. 136[3]	9230 C–2013[32]	B–0055–85[4].	
	Plate count	p. 143[3].			
6. Enterococci, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 139[3]	9230 B–2013.		
	MPN[6,8], multiple tube/multiple well, or.		9230 D–2013	D6503–99[9]	Enterolert®.[13,23]
	MF[2,5,6,7,8] single step or	1600[24]	9230 C–2013[32].		
	Plate count	p. 143[3].			
7. *Salmonella,* number per gram dry weight[11].	MPN multiple tube	1682[22].			
Aquatic Toxicity					
8. Toxicity, acute, fresh water organisms, LC_{50}, percent effluent.	*Ceriodaphnia dubia* acute	2002.0[25].			
	Daphnia puplex and *Daphnia magna* acute.	2021.0[25].			
	Fathead Minnow, *Pimephales promelas,* and Bannerfin shiner, *Cyprinella leedsi,* acute.	2000.0[25].			
	Rainbow Trout, *Oncorhynchus mykiss,* and brook trout, *Salvelinus fontinalis,* acute.	2019.0[25].			
9. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC_{50}, percent effluent.	Mysid, *Mysidopsis bahia,* acute	2007.0[25].			
	Sheepshead Minnow, *Cyprinodon variegatus,* acute.	2004.0[25].			
	Silverside, *Menidia beryllina, Menidia menidia,* and *Menidia peninsulae,* acute.	2006.0[25].			
10. Toxicity, chronic, fresh water organisms, NOEC or IC_{25}, percent effluent.	Fathead minnow, *Pimephales promelas,* larval survival and growth.	1000.0[26].			
	Fathead minnow, *Pimephales promelas,* embryo-larval survival and teratogenicity.	1001.0[26].			
	Daphnia, *Ceriodaphnia dubia,* survival and reproduction.	1002.0[26].			
	Green alga, *Selenastrum capricornutum,* growth.	1003.0[26].			
11. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC_{25}, percent effluent.	Sheepshead minnow, *Cyprinodon variegatus,* larval survival and growth.	1004.0[27].			
	Sheepshead minnow, *Cyprinodon variegatus,* embryo-larval survival and teratogenicity.	1005.0[27].			
	Inland silverside, *Menidia beryllina,* larval survival and growth.	1006.0[27].			
	Mysid, *Mysidopsis bahia,* survival, growth, and fecundity.	1007.0[27].			
	Sea urchin, *Arbacia punctulata,* fertilization.	1008.0[27].			

Table IA notes:

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

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

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

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

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

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

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

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

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

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

[11] Recommended for enumeration of target organism in sewage sludge.

[12] The multiple-tube fermentation test is used in 9221B.2–2014. 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.

[14] After prior enrichment in a presumptive medium for total coliform using 9221B.2–2014, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F–2014. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

[15] Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium, EPA–821–R–14–009. September 2014. 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® or 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.

[19] A description of the mColiBlue24® test is available from Hach Company.

[20] Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using A–1 Medium, EPA–821–R–06–013. July 2006. U.S. EPA.

[21] Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified Membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC), EPA–821–R–14–010. September 2014. U.S. EPA.

[22] Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium, EPA–821–R–14–012. September 2014. U.S. EPA.

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

[24] Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA–821–R–14–011. September 2014. U.S. EPA.

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

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

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

[28] To use Colilert-18® to assay for fecal coliforms, the incubation temperature is 44.5 ± 0.2 °C, and a water bath incubator is used.

[29] On a monthly basis, at least ten blue colonies from positive samples must be verified using Lauryl Tryptose Broth and EC broth, followed by count adjustment based on these results; and representative non-blue colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.

[30] On a monthly basis, at least ten sheen colonies from positive samples must be verified using lauryl tryptose broth and brilliant green lactose bile broth, followed by count adjustment based on these results; and representative non-sheen colonies should be verified using lauryl tryptose broth. Where possible, verifications should be done from randomized sample sources.

[31] Subject coliform positive samples determined by 9222 B–2015 or other membrane filter procedure to 9222 I–2015 using NA–MUG media.

[32] Verification of colonies by incubation of BHI agar at 10 ± 0.5 °C for 48 ± 3 h is optional. As per the Errata to the 23rd Edition of *Standard Methods for the Examination of Water and Wastewater* "Growth on a BHI agar plate incubated at 10 ± 0.5 °C for 48 ± 3 h is further verification that the colony belongs to the genus *Enterococcus*."

[33] 9221 F. 2–2014 This procedure allows for simultaneous detection of *E. coli* and thermotolerant coliforms by adding inverted vials to EC–MUG; the inverted vials collect gas produced by thermotolerant coliforms.

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
1. Acidity, as CaCO$_3$, mg/L.	Electrometric endpoint or phenolphthalein endpoint.		2310 B–2011	D1067–16	I–1020–85.[2]
2. Alkalinity, as CaCO$_3$, mg/L.	Electrometric or Colorimetric titration to pH 4.5, Manual.		2320 B–2011	D1067–16	973.43[3], I–1030–85.[2]
	Automatic	310.2 (Rev. 1974)[1]			I–2030–85.[2]
3. Aluminum—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 D–2011 or 3111 E–2011.		I–3051–85.[2]
	AA furnace		3113 B–2010.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	Direct Current Plasma (DCP)[36]			D4190–15	See footnote.[34]
	Colorimetric (Eriochrome cyanine R)		3500–Al B–2011.		
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–2011		973.49.[3]
	Nesslerization			D1426–15 (A)	973.49[3], I–3520–85.[2]
	Titration		4500–NH$_3$ C–2011.		
	Electrode		4500–NH$_3$ D–2011 or E–2011.	D1426–15 (B).	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction-based methods.		4500–NH_3 F–2011		See footnote.[60]
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction-based methods.	350.1[30], Rev. 2.0 (1993).	4500–NH_3 G–2011 4500–NH_3 H–2011		I–4523–85[2], I–2522–90.[80]
	Automated electrode				See footnote.[7]
	Ion Chromatography			D6919–17.	
	Automated gas diffusion, followed by conductivity cell analysis.				Timberline Ammonia–001.[74]
	Automated gas diffusion followed by fluorescence detector analysis.				FIAlab100.[82]
5. Antimony—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011.		
	AA furnace		3113 B–2010.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
6. Arsenic-Total,[4] mg/L	Digestion[4], followed by any of the following:.	206.5 (Issued 1978)[1].			
	AA gaseous hydride		3114 B–2011 or 3114 C–2011.	D2972–15 (B)	I–3062–85.[2]
	AA furnace		3113 B–2010	D2972–15 (C)	I–4063–98.[49]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4020–05.[70]
	Colorimetric (SDDC)		3500–As B–2011	D2972–15 (A)	I–3060–85.[2]
7. Barium-Total,[4] mg/L	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 D–2011		I–3084–85.[2]
	AA furnace		3113 B–2010	D4382–18.	
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	DCP[36]				See footnote.[34]
8. Beryllium—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration		3111 D–2011 or 3111 E–2011.	D3645–15 (A)	I–3095–85.[2]
	AA furnace		3113 B–2010	D3645–15 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	DCP			D4190–15	See footnote.[34]
	Colorimetric (aluminon)		See footnote[61].		
9. Biochemical oxygen demand (BOD_5), mg/L.	Dissolved Oxygen Depletion		5210 B–2016		973.44[3], p. 17[9], I–1578–78[8], See footnote.[10, 63]
10. Boron—Total,[37] mg/L.	Colorimetric (curcumin)		4500–B B–2011		I–3112–85.[2]
	ICP/AES	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP			D4190–15	See footnote.[34]
11. Bromide, mg/L	Electrode			D1246–16	I–1125–85.[2]
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B–2011, C–2011, D–2011.	D4327–17	993.30[3], I–2057–85.[79]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
12. Cadmium—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011 or 3111 C–2011.	D3557–17 (A or B)	974.27[3], p. 37[9], I–3135–85[2] or I–3136–85.[2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	AA furnace		3113 B–2010	D3557–17 (D)	I–4138–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–1472–85[2] or I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Voltametry[11]			D3557–17 (C).	
	Colorimetric (Dithizone)		3500–Cd–D–1990.		
13. Calcium—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration		3111 B–2011	D511–14 (B)	I–3152–85.[2]
	ICP/AES	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP				See footnote.[34]
	Titrimetric (EDTA)		3500–Ca B–2011	D511–14 (A).	
	Ion Chromatography			D6919–17.	
14. Carbonaceous biochemical oxygen demand ($CBOD_5$), mg/L[12].	Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B–2016		See footnote.[35, 63]
15. Chemical oxygen demand (COD), mg/L.	Titrimetric	410.3 (Rev. 1978)[1]	5220 B–2011 or C–2011.	D1252–06(12) (A)	973.46[3], p. 17[9], I–3560–85.[2]
	Spectrophotometric, manual or automatic	410.4, Rev. 2.0 (1993).	5220 D–2011	D1252–06(12) (B)	See footnotes[13, 14, 83], I–3561–85.[2]
16. Chloride, mg/L	Titrimetric: (silver nitrate)		4500–Cl^- B–2011	D512–12 (B)	I–1183–85.[2]
	(Mercuric nitrate)		4500–Cl^- C–2011	D512–12 (A)	973.51[3], I–1184–85.[2]
	Colorimetric: manual				I–1187–85.[2]
	Automated (ferricyanide)		4500–Cl^- E–2011		I–2187–85.[2]
	Potentiometric Titration		4500–Cl^- D–2011.		
	Ion Selective Electrode			D512–12 (C).	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B–2011 or 4110 C–2011.	D4327–17	993.30[3], I–2057–90.[51]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
17. Chlorine-Total residual, mg/L.	Amperometric direct		4500–Cl D–2011	D1253–14.	
	Amperometric direct (low level)		4500–Cl E–2011.		
	Iodometric direct		4500–Cl B–2011.		
	Back titration ether end-point[15]		4500–Cl C–2011.		
	DPD–FAS		4500–Cl F–2011.		
	Spectrophotometric, DPD		4500–Cl G–2011.		
	Electrode				See footnote.[16]
17A. Chlorine-Free Available, mg/L.	Amperometric direct		4500–Cl D–2011	D1253–14.	
	Amperometric direct (low level)		4500–Cl E–2011.		
	DPD–FAS		4500–Cl F–2011.		
	Spectrophotometric, DPD		4500–Cl G–2011.		
18. Chromium VI dissolved, mg/L.	0.45-micron filtration followed by any of the following:				
	AA chelation-extraction		3111 C–2011		I–1232–85.[2]
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500–Cr C–2011	D5257–17	993.23.[3]
	Colorimetric (diphenyl-carbazide)		3500–Cr B–2011	D1687–17 (A)	I–1230–85.[2]
19. Chromium—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011	D1687–17 (B)	974.27[3], I–3236–85.[2]
	AA chelation-extraction		3111 C–2011.		
	AA furnace		3113 B–2010	D1687–17 (C)	I–3233–93.[46]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68], 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4020–05[70] I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Colorimetric (diphenyl-carbazide)		3500–Cr B–2011.		
20. Cobalt—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration		3111 B–2011 or 3111 C–2011.	D3558–15 (A or B)	p. 37[9], I–3239–85.[2]
	AA furnace		3113 B–2010	D3558–15 (C)	I–4243–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4020–05[70] I–4472–97.[81]
	DCP			D4190–15	See footnote.[34]
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity.	Colorimetric (ADMI)		2120 F–2011[78].		
	Platinum cobalt visual comparison		2120 B–2011		I–1250–85.[2]
	Spectrophotometric				See footnote.[18]
22. Copper—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011 or 3111 C–2011.	D1688–17 (A or B)	974.27[3], p. 37[9], I–3270–85[2] or I–3271–85.[2]
	AA furnace		3113 B–2010	D1688–17 (C)	I–4274–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4020–05[70], I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Colorimetric (Neocuproine)		3500–Cu B–2011.		
	Colorimetric (Bathocuproine)		3500–Cu C–2011		See footnote.[19]
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.			D7511–12(17).	
	Manual distillation with $MgCl_2$, followed by any of the following:	335.4, Rev. 1.0 (1993)[57].	4500–CN^- B–2016 and C–2016.	D2036–09(15)(A), D7284–13(17).	10–204–00–1–X.[56]
	Flow Injection, gas diffusion amperometry			D2036–09(15)(A) D7284–13(17).	
	Titrimetric		4500–CN^- D–2016	D2036–09(15)(A)	p. 22.[9]
	Spectrophotometric, manual		4500–CN^- E–2016	D2036–09(15)(A)	I–3300–85.[2]
	Semi-Automated[20]	335.4, Rev. 1.0 (1993)[57].	4500–CN^- N–2016		10–204–00–1–X[56], I–4302–85.[2]
	Ion Chromatography			D2036–09(15)(A).	
	Ion Selective Electrode		4500–CN^- F–2016	D2036–09(15)(A).	
24. Cyanide-Available, mg/L.	Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$, followed by Titrimetric or Spectrophotometric.		4500–CN^- G–2016	D2036–09(15)(B).	
	Flow injection and ligand exchange, followed by gas diffusion amperometry.[59]			D6888–16	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.			D7237–15 (A)	OIA–1677–09.[44]
	Manual micro-diffusion and colorimetry			D4282–15.	
25. Fluoride—Total, mg/L.	Manual distillation[6], followed by any of the following:.		4500–F^- B–2011.		
	Electrode, manual		4500–F^- C–2011	D1179–16 (B).	
	Electrode, automated				I–4327–85.[2]
	Colorimetric, (SPADNS)		4500–F^- D–2011	D1179–16 (A).	
	Automated complexone		4500–F^- E–2011.		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B–2011 or C–2011.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
26. Gold—Total,[4] mg/L	Digestion[4], followed by any of the following:				
	AA direct aspiration		3111 B–2011.		
	AA furnace	231.2 (Issued 1978)[1]	3113 B–2010.		
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP				See footnote.[34]
27. Hardness—Total, as $CaCO_3$, mg/L.	Automated colorimetric	130.1 (Issued 1971)[1].			
	Titrimetric (EDTA)		2340 C–2011	D1126–17	973.52B[3], I–1338–85.[2]
	Ca plus Mg as their carbonates, by any approved method for Ca and Mg (See Parameters 13 and 33), provided that the sum of the lowest point of quantitation for Ca and Mg is below the NPDES permit requirement for Hardness.		2340 B–2011.		
28. Hydrogen ion (pH), pH units.	Electrometric measurement		4500–H^+ B–2011	D1293–99 (A or B)	973.41[3], I–1586–85.[2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	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		3111 B–2011.		
	AA furnace	235.2 (Issued 1978)[1].			
	ICP/MS		3125 B–2011.		
30. Iron—Total,[4] mg/L	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011 or 3111 C–2011.	D1068–15 (A)	974.27[3], I–3381–85.[2]
	AA furnace		3113 B–2010	D1068–15 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP[36]			D4190–15	See footnote.[34]
	Colorimetric (Phenanthroline)		3500-Fe B–2011	D1068–15 (C)	See footnote.[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–2011 or C–2011 and 4500–NH_3 B–2011.	D3590–17 (A)	I–4515–91.[45]
	Titration		4500–NH_3 C–2011		973.48.[3]
	Nesslerization			D1426–15 (A).	
	Electrode		4500–NH_3 D–2011 or E–2011.	D1426–15 (B).	
	Semi-automated phenate	350.1, Rev. 2.0 (1993).	4500–NH_3 G–2011 4500–NH_3 H–2011.		
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.		4500–NH_3 F–2011		See footnote.[60]
	Automated gas diffusion, followed by conductivity cell analysis.				Timberline Ammonia-001.[74]
	Automated gas diffusion followed by fluorescence detector analysis.				FIAlab 100.[82]
	Automated Methods for TKN that do not require manual distillation.				
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation).	351.1 (Rev. 1978)[1]			I–4551–78.[8]
	Semi-automated block digestor colorimetric (distillation not required).	351.2, Rev. 2.0 (1993).	4500–N_{org} D–2011	D3590–17 (B)	I–4515–91.[45]
	Block digester, followed by Auto distillation and Titration.				See footnote.[39]
	Block digester, followed by Auto distillation and Nesslerization.				See footnote.[40]
	Block Digester, followed by Flow injection gas diffusion (distillation not required).				See footnote.[41]
	Digestion with peroxdisulfate, followed by Spectrophotometric (2,6-dimethyl phenol).				Hach 10242.[76]
	Digestion with persulfate, followed by Colorimetric.				NCASI TNTP W10900.[77]
32. Lead—Total,[4] mg/L	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011 or 3111 C–2011..	D3559–15 (A or B)	974.27[3], I–3399–85.[2]
	AA furnace		3113 B–2010	D3559–15 (D)	I–4403–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Voltametry[11]			D3559–15 (C).	
	Colorimetric (Dithizone)		3500-Pb B–2011		
33. Magnesium—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration		3111 B–2011	D511–14 (B)	974.27[3], I–3447–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP				See footnote.[34]
	Ion Chromatography			D6919–17.	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
34. Manganese—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011	D858–17 (A or B)	974.27[3], I–3454–85.[2]
	AA furnace		3113 B–2010	D858–17 (C).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Colorimetric (Persulfate)		3500-Mn B–2011		920.203.[3]
	Colorimetric (Periodate)				See footnote.[23]
35. Mercury—Total, mg/L.	Cold vapor, Manual	245.1, Rev. 3.0 (1994).	3112 B–2011	D3223–17	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		3111 D–2011		I–3490–85.[2]
	AA furnace		3113 B–2010		I–3492–96.[47]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4472–97.[81]
	DCP				See footnote.[34]
37. Nickel—Total,[4] mg/L.	Digestion[4], followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011 or 3111 C–2011	D1886–14 (A or B)	I–3499–85.[2]
	AA furnace		3113 B–2010	D1886–14 (C)	I–4503–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14[3], I–4020–05[70] I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
38. Nitrate (as N), mg/L	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C–2011.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
	Ion Selective Electrode		4500–NO_3^- D–2016.		
	Colorimetric (Brucine sulfate)	352.1 (Issued 1971)[1]			973.50[3], 419D[17], p. 28.[9]
	Spectrophotometric (2,6-dimethylphenol)				Hach 10206.[75]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40)				
39. Nitrate-nitrite (as N), mg/L.	Cadmium reduction, Manual		4500–NO_3^- E–2016	D3867–16 (B).	
	Cadmium reduction, Automated	353.2, Rev. 2.0 (1993).	4500–NO_3^- F–2016 4500–NO_3^- I–2016	D3867–16 (A)	I–2545–90.[51]
	Automated hydrazine		4500–NO_3^- H–2016.		
	Reduction/Colorimetric				See footnote.[62]
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C–2011.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
	Enzymatic reduction, followed by automated colorimetric determination.			D7781–14	I–2547–11[72] I–2548–11[72] N07–0003.[73]
	Enzymatic reduction, followed by manual colorimetric determination.		4500–NO_3^- J–2018.		
	Spectrophotometric (2,6-dimethylphenol)				Hach 10206.[75]
40. Nitrite (as N), mg/L	Spectrophotometric: Manual		4500–NO_2^- B–2011		See footnote.[25]
	Automated (Diazotization)				I–4540–85[2], See footnote[62] I–2540–90.[80]
	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993).	4500–NO_3^- F–2016 4500–NO_3^- I–2016	D3867–16 (A)	I–4545–85.[2]
	Manual (*bypass cadmium or enzymatic reduction).		4500–NO_3^- E–2016, 4500–NO_3 J–2018.	D3867–16 (B).	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C–2011.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	EPA [52]	Standard methods [84]	ASTM	USGS/AOAC/other
	Automated (* bypass Enzymatic reduction)			D7781–14	I–2547–11 [72] I–2548–11 [72] N07–0003.[73]
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–2011 [38].		
	Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry.	1664 Rev. A; 1664 Rev. B [42].	5520 B–2011 [38] and 5520 F–2011 [38].		
42. Organic carbon—Total (TOC), mg/L.	Combustion		5310 B–2014	D7573–09(17)	973.47 [3], p. 14.[24]
	Heated persulfate or UV persulfate oxidation.		5310 C–2014 5310 D–2011	D4839–03(17)	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.	Ascorbic acid method:				
	Automated	365.1, Rev. 2.0 (1993).	4500–P F–2011 or G–2011.		973.56 [3], I–4601–85 [2], I–2601–90.[80]
	Manual, single-reagent		4500–P E–2011	D515–88 (A)	973.55.[3]
	Manual, two-reagent	365.3 (Issued 1978) [1].			
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C–2011.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
45. Osmium—Total [4], mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration		3111 D–2011.		
	AA furnace	252.2 (Issued 1978) [1].			
46. Oxygen, dissolved, mg/L.	Winkler (Azide modification)		4500–O (B–F)-2016	D888–12 (A)	973.45B [3], I–1575–78.[8]
	Electrode		4500–O G–2016	D888–12 (B)	I–1576–78.[8]
	Luminescence-Based Sensor		4500–O H–2016	D888–12 (C)	See footnote.[63] See footnote.[64]
47. Palladium—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration		3111 B–2011.		
	AA furnace	253.2 (Issued 1978) [1].			
	ICP/MS		3125 B–2011.		
	DCP				See footnote.[34]
48. Phenols, mg/L	Manual distillation [26], followed by any of the following:	420.1 (Rev. 1978) [1]	5530 B–2010	D1783–01(12)	
	Colorimetric (4AAP) manual	420.1 (Rev. 1978) [1]	5530 D–2010 [27]	D1783–01(12) (A or B)	
	Automated colorimetric (4AAP)	420.4 Rev. 1.0 (1993).			
49. Phosphorus (elemental), mg/L.	Gas-liquid chromatography				See footnote.[28]
50. Phosphorus—Total, mg/L.	Digestion [20], followed by any of the following:		4500–P B(5)–2011		973.55.[3]
	Manual	365.3 (Issued 1978) [1]	4500–P E–2011	D515–88 (A)	
	Automated ascorbic acid reduction	365.1 Rev. 2.0 (1993).	4500–P (F–H)–2011		973.56 [3], I–4600–85.[2]
	ICP/AES [4] [36]	200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97.[50]
	Semi-automated block digestor (TKP digestion).	365.4 (Issued 1974) [1]		D515–88 (B)	I–4610–91.[48]
	Digestion with persulfate, followed by Colorimetric.				NCASI TNTP W10900.[77]
51. Platinum—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration		3111 B–2011.		
	AA furnace	255.2 (Issued 1978) [1].			
	ICP/MS		3125 B–2011.		
	DCP				See footnote.[34]
52. Potassium—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration		3111 B–2011		973.53 [3], I–3630–85.[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2011.		
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	Flame photometric		3500–K B–2011.		
	Electrode		3500–K C–2011.		
	Ion Chromatography			D6919–17.	
53. Residue—Total, mg/L.	Gravimetric, 103–105°		2540 B–2015		I–3750–85.[2]
54. Residue—filterable, mg/L.	Gravimetric, 180°		2540 C–2015	D5907–13	I–1750–85.[2]
55. Residue—non-filterable (TSS), mg/L.	Gravimetric, 103–105° post-washing of residue.		2540 D–2015	D5907–13	I–3765–85.[2]
56. Residue—settleable, ml/L.	Volumetric (Imhoff cone), or gravimetric		2540 F–2015.		

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	EPA [52]	Standard methods [84]	ASTM	USGS/AOAC/other
57. Residue—Volatile, mg/L.	Gravimetric, 550°	160.4 (Issued 1971) [1]	2540 E–2015		I–3753–85.[2]
58. Rhodium—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration, or		3111 B–2011.		
	AA furnace	265.2 (Issued 1978) [1].			
	ICP/MS		3125 B–2011.		
59. Ruthenium—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration, or		3111 B–2011.		
	AA furnace	267.2 [1].			
	ICP/MS		3125 B–2011.		
60. Selenium—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA furnace		3113 B–2010	D3859–15 (B)	I–4668–98.[49]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES [36]	200.5, Rev 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14 [3], I–4020–05 [70] I–4472–97.[81]
	AA gaseous hydride		3114 B–2011, or 3114 C–2011.	D3859–15 (A)	I–3667–85.[2]
61. Silica—Dissolved,[37] mg/L.	0.45-micron filtration followed by any of the following:				
	Colorimetric, Manual		4500-SiO_2 C–2011	D859–16	I–1700–85.[2]
	Automated (Molybdosilicate)		4500-SiO_2 E–2011 or F–2011.		I–2700–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
62. Silver—Total,[4, 31] mg/L.	Digestion [4 29], followed by any of the following:				
	AA direct aspiration		3111 B–2011 or 3111 C–2011.		974.27 [3], p. 37 [9], I–3720–85.[2]
	AA furnace		3113 B–2010		I–4724–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14 [3], I–4472–97.[81]
	DCP				See footnote.[34]
63. Sodium—Total,[4] mg/L.	Digestion [4], followed by any of the following:				
	AA direct aspiration		3111 B–2011		973.54 [3], I–3735–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003) [68]; 200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP				See footnote.[34]
	Flame photometric		3500-Na B–2011.		
	Ion Chromatography			D6919–17.	
64. Specific conductance, micromhos/cm at 25 °C.	Wheatstone bridge	120.1 (Rev. 1982) [1]	2510 B–2011	D1125–95(99) (A)	973.40,[3] I–2781–85.[2]
65. Sulfate (as SO_4), mg/L.	Automated colorimetric	375.2, Rev. 2.0 (1993).	4500–SO_4^2 F–2011 or G–2011.		
	Gravimetric		4500–SO_4^2 C–2011 or D–2011.		925.54.[3]
	Turbidimetric		4500–SO_4^2 E–2011.	D516–16.	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C–2011.	D4327–17	993, I–4020–05 [70].30$_3$
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2.[54]
66. Sulfide (as S), mg/L	Sample Pretreatment		4500–S^{2-} B, C–2011.		
	Titrimetric (iodine)		4500–S^{2-} F–2011		I–3840–85.[2]
	Colorimetric (methylene blue)		4500–S^{2-} D–2011.		
	Ion Selective Electrode		4500–S^{2-} G–2011	D4658–15.	
67. Sulfite (as SO_3), mg/L.	Titrimetric (iodine-iodate)		4500–SO_3^{2-} B–2011.		
68. Surfactants, mg/L	Colorimetric (methylene blue)		5540 C–2011	D2330–02.	
69. Temperature, °C	Thermometric		2550 B–2010		See footnote.[32]
70. Thallium-Total,[4] mg/L.	Digestion,[4] followed by any of the following:				

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	AA direct aspiration		3111 B–2011.		
	AA furnace	279.2 (Issued 1978)[1].	3113 B–2010.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14,[3] I–4471–97[50] I–4472–97.[81]
71. Tin-Total,[4] mg/L	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 B–2011		I–3850–78.[8]
	AA furnace		3113 B–2010.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).			
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
72. Titanium-Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 D–2011.		
	AA furnace	283.2 (Issued 1978)[1].			
	ICP/AES	200.7, Rev. 4.4 (1994).			
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14.[3]
	DCP				See footnote.[34]
73. Turbidity, NTU[53]	Nephelometric	180.1, Rev. 2.0 (1993).	2130 B–2011	D1889–00	I–3860–85[2] See footnote[65] See footnote[66] See footnote.[67]
74. Vanadium-Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 D–2011.		
	AA furnace		3113 B–2010	D3373–17.	
	ICP/AES	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14,[3] I–4020–05.[70]
	DCP			D4190–15	See footnote.[34]
	Colorimetric (Gallic Acid)		3500–V B–2011.		
75. Zinc-Total[4], mg/L	Digestion,[4] followed by any of the following:				
	AA direct aspiration[36]		3111 B–2011 or 3111 C–2011.	D1691–17 (A or B)	974.27,[3] p. 37,[9] I–3900–85.[2]
	AA furnace	289.2 (Issued 1978)[1].			
	ICP/AES[36]	200.5, Rev. 4.2 (2003)[68]; 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14,[3] I–4020–05[70] I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Colorimetric (Zincon)		3500 Zn B–2011		See footnote.[33]
76. Acid Mine Drainage		1627[69].			

Table IB Notes:

[1] Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020. Revised March 1983 and 1979, where applicable. U.S. EPA.

[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 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 (FLAA) determinations, 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 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 (for 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.

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

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

[12] Carbonaceous biochemical oxygen demand (CBOD$_5$) must not be confused with the traditional BOD$_5$ test method which measures ‘‘total 5-day BOD.’’ The addition of the nitrification inhibitor is not a procedural option but must be included to report the CBOD$_5$ parameter. A discharger whose permit requires reporting the traditional BOD$_5$ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD$_5$ is required can the permittee report data using a nitrification inhibitor.

[13] OIC Chemical Oxygen Demand Method. 1978. Oceanography International Corporation.

[14] Method 8000, Chemical Oxygen Demand, Hach Handbook of Water Analysis, 1979. Hach Company.

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

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

[17] Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, EPA–821–R–05–001. Revision 2.0, February 2005. US EPA.

[18] National Council of the Paper Industry for Air and Stream Improvement (NCASI) Technical Bulletin 253 (1971) and Technical Bulletin 803, May 2000.

[19] Method 8506, Bicinchoninate Method for Copper, Hach Handbook of Water Analysis. 1979. Hach Company.

[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.

[22] Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water. 1980. Hach Company.

[23] Method 8034, Periodate Oxidation Method for Manganese, Hach Handbook of Wastewater Analysis. 1979. Hach Company.

[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). 1987. USGS.

[25] Method 8507, Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater. 1979. Hach Company.

[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.

[29] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

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

[31] For samples known or suspected to contain high levels of silver (*e.g.,* in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH_4OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I_2 to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH >7 with NH_4OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.

[32] ‘‘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.

[33] Method 8009, Zincon Method for Zinc, Hach Handbook of Water Analysis, 1979. Hach Company.

[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.

[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

[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.

[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.

[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.

[43] Method 1631, Revision E, 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.

[44] Method OIA–1677–09, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA). 2010. OI Analytical.

[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, 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 U.S. EPA. Revision 1.0, 1997, including errata cover sheet April 27, 1999. Determination of Inorganic Ions in Drinking Water by Ion Chromatography.

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

[54] Method D6508–15, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. 2015. ASTM

[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. 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.

[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 D6888–16 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 for the Examination of Water and Wastewater* may be used. This method has poorer precision and bias than the methods of choice.

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

[63] Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD$_5$ and CBOD$_5$. Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).

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

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

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

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

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

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

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

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[72] USGS Techniques and Methods 5–B8, Chapter 8, Section B, Methods of the National Water Quality Laboratory Book 5, Laboratory Analysis, 2011 USGS

[73] NECi Method N07–0003, ’’Nitrate Reductase Nitrate-Nitrogen Analysis,’’ Revision 9.0, March 2014, The Nitrate Elimination Co., Inc.

[74] Timberline Instruments, LLC Method Ammonia-001, ‘‘Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Conductivity Cell Analysis,’’ June 2011, Timberline Instruments, LLC.

[75] Hach Company Method 10206, ‘‘Spectrophotometric Measurement of Nitrate in Water and Wastewater,’’ Revision 2.1, January 2013, Hach Company.

[76] Hach Company Method 10242, ‘‘Simplified Spectrophotometric Measurement of Total Kjeldahl Nitrogen in Water and Wastewater,’’ Revision 1.1, January 2013, Hach Company.

[77] National Council for Air and Stream Improvement (NCASI) Method TNTP–W10900, ‘‘Total (Kjeldahl) Nitrogen and Total Phosphorus in Pulp and Paper Biologically Treated Effluent by Alkaline Persulfate Digestion,’’ June 2011, National Council for Air and Stream Improvement, Inc.

[78] The pH adjusted sample is to be adjusted to 7.6 for NPDES reporting purposes.

[79] I–2057–85 U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Chap. A11989, Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, 1989.

[80] Methods I–2522–90, I–2540–90, and I–2601–90 U.S. Geological Survey 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.

[81] Method I–1472–97, U.S. Geological Survey Open-File Report 98–165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory–Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments, 1998.

[82] FIAlab Instruments, Inc. Method FIAlab 100, ‘‘Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Fluorescence Detector Analysis’’, April 4, 2018, FIAlab Instruments, Inc.

[83] MACHEREY–NAGEL GmbH and Co. Method 036/038 NANOCOLOR® COD LR/HR, ‘‘Spectrophotometric Measurement of Chemical Oxygen Demand in Water and Wastewater’’, Revision 1.5, May 2008, MACHEREY–NAGEL GmbH and Co. KG.

[84] Please refer to the following applicable Quality Control Sections: Part 2000 Methods, Physical and Aggregate Properties 2020 (2017); Part 3000 Methods, Metals, 3020 (2017); Part 4000 Methods, Inorganic Nonmetallic Constituents, 4020 (2014); Part 5000 Methods, and Aggregate Organic Constituents, 5020 (2017). These Quality Control Standards are available for download at *www.standardmethods.org* at no charge.

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

Parameter[1]	Method	EPA[2][7]	Standard methods	ASTM	Other
1. Acenaphthene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
2. Acenaphthylene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
3. Acrolein	GC	603.			
	GC/MS	624.1[4], 1624B.			
4. Acrylonitrile	GC	603.			
	GC/MS	624.1[4], 1624B			O–4127–96.[13]
5. Anthracene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440B–2005	D4657–92 (98).	
6. Benzene	GC	602	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
7. Benzidine	Spectro-photometric.				See footnote[3], p. 1.
	GC/MS	625.1[5], 1625B	6410 B–2000.		
	HPLC	605.			
8. Benzo(a)anthracene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
9. Benzo(a)pyrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
10. Benzo(b)fluoranthene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
11. Benzo(g,h,i)perylene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
12. Benzo(k)fluoranthene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
13. Benzyl chloride	GC				See footnote[3], p. 130.
	GC/MS				See footnote[6], p. S102.

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

Parameter [1]	Method	EPA [2,7]	Standard methods	ASTM	Other
14. Butyl benzyl phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
15. bis(2-Chloroethoxy) methane	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
16. bis(2-Chloroethyl) ether	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
17. bis(2-Ethylhexyl) phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
18. Bromodichloromethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
19. Bromoform	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
20. Bromomethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
21. 4-Bromophenyl phenyl ether	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
22. Carbon tetrachloride	GC	601	6200 C–2011		See footnote [3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
23. 4-Chloro-3-methyl phenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
24. Chlorobenzene	GC	601, 602	6200 C–2011		See footnote [3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
25. Chloroethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96.[13]
26. 2-Chloroethylvinyl ether	GC	601.			
	GC/MS	624.1, 1624B.			
27. Chloroform	GC	601	6200 C–2011		See footnote [3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
28. Chloromethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
29. 2-Chloronaphthalene	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
30. 2-Chlorophenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
31. 4-Chlorophenyl phenyl ether	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
32. Chrysene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
33. Dibenzo(a,h)anthracene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
34. Dibromochloromethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
35. 1,2-Dichlorobenzene	GC	601, 602	6200 C–2011.		
	GC/MS	624.1, 1625B	6200 B–2011		See footnote [9], p. 27; O–4127–96 [13], O–4436–16.[14]
36. 1,3-Dichlorobenzene	GC	601, 602	6200 C–2011.		
	GC/MS	624.1, 1625B	6200 B–2011		See footnote [9], p. 27; O–4127–96.[13]
37. 1,4-Dichlorobenzene	GC	601, 602	6200 C–2011.		
	GC/MS	624.1, 1625B	6200 B–2011		See footnote [9], p. 27; O–4127–96 [13], O–4436–16.[14]
38. 3,3′-Dichlorobenzidine	GC/MS	625.1, 1625B	6410 B–2000.		
	HPLC	605.			
39. Dichlorodifluoromethane	GC	601.			
	GC/MS		6200 C–2011		O–4127–96 [13], O–4436–16.[14]
40. 1,1-Dichloroethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
41. 1,2-Dichloroethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
42. 1,1-Dichloroethene	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
43. *trans*-1,2-Dichloroethene	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
44. 2,4-Dichlorophenol	GC	604	6420 B–2000..		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
45. 1,2-Dichloropropane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
46. *cis*-1,3-Dichloropropene	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
47. *trans*-1,3-Dichloropropene	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
48. Diethyl phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
49. 2,4-Dimethylphenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
50. Dimethyl phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
51. Di-*n*-butyl phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
52. Di-*n*-octyl phthalate	GC	606.			

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

Parameter [1]	Method	EPA [2] [7]	Standard methods	ASTM	Other
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
53. 2,4-Dinitrophenol	GC	604	6420 B–2000		See footnote [9], p. 27.
	GC/MS	625.1, 1625B	6410 B–2000.		
54. 2,4-Dinitrotoluene	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
55. 2,6-Dinitrotoluene	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
56. Epichlorohydrin	GC				See footnote [3], p. 130.
	GC/MS				See footnote [6], p. S102.
57. Ethylbenzene	GC	602	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
58. Fluoranthene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
59. Fluorene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran.	GC/MS	1613B.			
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran.	GC/MS	1613B.			
62. 1,2,3,4,6,7,8-Heptachloro-dibenzo-*p*-dioxin.	GC/MS	1613B.			
63. Hexachlorobenzene	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
64. Hexachlorobutadiene	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27; O–4127–96.[13]
65. Hexachlorocyclopentadiene	GC	612.			
	GC/MS	625.1 [5], 1625B	6410 B–2000		See footnote [9], p. 27; O–4127–96.[13]
66. 1,2,3,4,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B.			
67. 1,2,3,6,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B.			
68. 1,2,3,7,8,9-Hexachloro-dibenzofuran.	GC/MS	1613B.			
69. 2,3,4,6,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B.			
70. 1,2,3,4,7,8-Hexachloro-dibenzo-*p*-dioxin.	GC/MS	1613B.			
71. 1,2,3,6,7,8-Hexachloro-dibenzo-*p*-dioxin.	GC/MS	1613B.			
72. 1,2,3,7,8,9-Hexachloro-dibenzo-*p*-dioxin.	GC/MS	1613B.			
73. Hexachloroethane	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27; O–4127–96.[13]
74. Indeno(1,2,3-c,d) pyrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
75. Isophorone	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
76. Methylene chloride	GC	601	6200 C–2011		See footnote [3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 [13], O–4436–16.[14]
77. 2-Methyl-4,6-dinitrophenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
78. Naphthalene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC	610	6440 B–2005.		
79. Nitrobenzene	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
	HPLC			D4657–92 (98).	
80. 2-Nitrophenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
81. 4-Nitrophenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
82. N-Nitrosodimethylamine	GC	607.			
	GC/MS	625.1 [5], 1625B	6410 B–2000		See footnote [9], p. 27.
83. N-Nitrosodi-*n*-propylamine	GC	607.			
	GC/MS	625.1 [5], 1625B	6410 B–2000		See footnote [9], p. 27.
84. N-Nitrosodiphenylamine	GC	607.			
	GC/MS	625.1 [5], 1625B	6410 B–2000		See footnote [9], p. 27.
85. Octachlorodibenzofuran	GC/MS	1613B [10].			
86. Octachlorodibenzo-*p*-dioxin	GC/MS	1613B [10].			
87. 2,2′-oxybis(1-chloropropane) [12] [also known as bis(2-Chloro-1-methylethyl) ether].	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote [9], p. 27.
88. PCB–1016	GC	608.3			See footnote [3], p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2000.		
89. PCB–1221	GC	608.3			See footnote [3], p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2000.		
90. PCB–1232	GC	608.3			See footnote [3], p. 43; See footnote.[8]

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

Parameter[1]	Method	EPA[2 7]	Standard methods	ASTM	Other
	GC/MS	625.1	6410 B–2000.		
91. PCB–1242	GC	608.3			See footnote[3], p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2000.		
92. PCB–1248	GC	608.3			See footnote[3], p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2000.		
93. PCB–1254	GC	608.3			See footnote[3], p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2000.		
94. PCB–1260	GC	608.3			See footnote[3], p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2000.		
95. 1,2,3,7,8-Pentachloro-dibenzofuran.	GC/MS	1613B.			
96. 2,3,4,7,8-Pentachloro-dibenzofuran.	GC/MS	1613B.			
97. 1,2,3,7,8-Pentachloro-dibenzo-*p*-dioxin.	GC/MS	1613B.			
98. Pentachlorophenol	GC	604	6420 B–2000		See footnote[3], p. 140.
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
99. Phenanthrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
100. Phenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
101. Pyrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98).	
102. 2,3,7,8-Tetrachloro-dibenzofuran	GC/MS	1613B[10].			
103. 2,3,7,8-Tetrachloro-dibenzo-*p*-dioxin.	GC/MS	613, 625.1[5a], 1613B.			
104. 1,1,2,2-Tetrachloroethane	GC	601	6200 C–2011		See footnote[3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96.[13]
105. Tetrachloroethene	GC	601	6200 C–2011		See footnote[3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
106. Toluene	GC	602	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
107. 1,2,4-Trichlorobenzene	GC	612			See footnote[3], p. 130.
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27; O–4127–96[13], O–4436–16.[14]
108. 1,1,1-Trichloroethane	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
109. 1,1,2-Trichloroethane	GC	601	6200 C–2011		See footnote[3], p. 130.
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
110. Trichloroethene	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
111. Trichlorofluoromethane	GC	601	6200 C–2011.		
	GC/MS	624.1	6200 B–2011		O–4127–96.[13]
112. 2,4,6-Trichlorophenol	GC	604	6420 B–2000.		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote[9], p. 27.
113. Vinyl chloride	GC	601	6200 C–2011.		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96[13], O–4436–16.[14]
114. Nonylphenol	GC/MS			D7065–17.	
115. Bisphenol A (BPA)	GC/MS			D7065–17.	
116. *p-tert*-Octylphenol (OP)	GC/MS			D7065–17.	
117. Nonylphenol Monoethoxylate (NP1EO).	GC/MS			D7065–17.	
118. Nonylphenol Diethoxylate (NP2EO).	GC/MS			D7065–17.	
119. Adsorbable Organic Halides (AOX).	Adsorption and Coulometric Titration.	1650[11].			
120. Chlorinated Phenolics	In Situ Acetylation and GC/MS.	1653[11].			

Table IC notes:

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

[2] The full text of Methods 601–613, 1613B, 1624B, and 1625B are provided at appendix A, Test Procedures for Analysis of Organic Pollutants. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B of this part, Definition and Procedure for the Determination of the Method Detection Limit. These methods are available at: *https://www.epa.gov/cwa-methods* as individual PDF files.

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

[4] Method 624.1 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 624.1.

[5] Method 625.1 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 625.1 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, 1624B, and 1625B in accordance with procedures in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624.1 and 625.1 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 quality control (QC) acceptance criteria in the pertinent method, analytical results for that parameter in the unspiked sample are suspect. The results should be reported but cannot be used to demonstrate regulatory compliance. If the method does not contain QC acceptance criteria, control limits of ± three standard deviations around the mean of a minimum of five replicate measurements must be used. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

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

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

[10] Analysts may use Fluid Management Systems, Inc. Power-Prep system in place of manual cleanup provided the analyst meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities. Method 1613, Revision B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. Revision B, 1994. U.S. EPA. The full text of this method is provided in appendix A to this part and at *https://www.epa.gov/cwa-methods/approved-cwa-test-methods-organic-compounds.*

[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 in part 430 of this chapter, The Pulp, Paper, and Paperboard Point Source Category.

[12] The compound was formerly inaccurately labeled as 2,2′-oxybis(2-chloropropane) and bis(2-chloroisopropyl) ether. Some versions of Methods 611, and 1625 inaccurately list the analyte as ''bis(2-chloroisopropyl)ether,'' but use the correct CAS number of 108–60–1.

[13] Method O–4127–96, U.S. Geological Survey Open-File Report 97–829, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of 86 volatile organic compounds in water by gas chromatography/mass spectrometry, including detections less than reporting limits,1998, USGS.

[14] Method O–4436–16 U.S. Geological Survey Techniques and Methods, book 5, chap. B12, Determination of heat purgeable and ambient purgeable volatile organic compounds in water by gas chromatography/mass spectrometry, 2016, USGS.

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TABLE IH—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.	Most Probable Number (MPN), 5 tube, 3 dilution, or.	p. 132[3]	9221 E–2014, 9221 F.2–2014[32].		
	Membrane filter (MF)[2], single step.	p. 124[3]	9222 D–2015[26]	B–0050–85[4].	
2. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 114[3]	9221 B–2014.		
	MF[2], single step or two step.	p. 108[3]	9222 B–2015[27]	B–0025–85[4].	
	MF[2] with enrichment	p. 111[3]	9222 (B + B.4e)—2015[27].		
3. *E. coli,* number per 100 mL.	MPN[5 7 13], multiple tube, or.		9221 B.3–2014/9221 F–2014[10 12 32].		
	Multiple tube/multiple well, or.		9223 B–2016[11]	991.15[9]	Colilert®[11 15], Colilert-18®.[11 14 15]
	MF[2 5 6 7], two step, or	1103.1[18]	9222 B–2015/9222 I–2015[17], 9213 D–2007.	D5392–93[8].	
	Single step	1603[19], 1604[20]			m-ColiBlue24®[16], KwikCount™ EC.[28 29]
4. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 139[3]	9230 B–2013.		
	MF[2], or	p. 136[3]	9230 C–2013[30]	B–0055–85[4].	
	Plate count	p. 143[3].			
5. Enterococci, number per 100 mL.	MPN[5 7], multiple tube/multiple well, or.		9230 D–2013	D6503–99[8]	Enterolert®.[11 21]
	MF[2 5 6 7] two step, or	1106.1[22]	9230 C–2013[30]	D5259–92[8].	
	Single step, or	1600[23]	9230 C–2013[30].		
	Plate count	p. 143[3].			
Protozoa					
6. *Cryptosporidium*	Filtration/IMS/FA	1622[24], 1623[25], 1623.1[25 31].			
7. *Giardia*	Filtration/IMS/FA	1623[25], 1623.1[25 31].			

Table 1H notes:

[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] 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.

[6] 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.

[7] 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.

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

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

[10] The multiple-tube fermentation test is used in 9221B.3–2014. 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.

[11] These tests are collectively known as defined enzyme substrate tests.

[12] After prior enrichment in a presumptive medium for total coliform using 9221B.3–2014, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F–2014. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

[13] 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.

[14] 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.

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

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

[17] Subject coliform positive samples determined by 9222B–2015 or other membrane filter procedure to 9222I–2015 using NA–MUG media.

[18] 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.

[19] Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC), EPA–821–R–14–010. September 2014. US EPA.

[20] 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.

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

[22] 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.

[23] Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA–821–R–14–011. September 2014. US EPA.

[24] 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.

[25] Methods 1623 and 1623.1 use 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. Method 1623.1: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. EPA 816–R–12–001. January 2012. US EPA.

[26] On a monthly basis, at least ten blue colonies from positive samples must be verified using Lauryl Tryptose Broth and EC broth, followed by count adjustment based on these results; and representative non-blue colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.

[27] On a monthly basis, at least ten sheen colonies from positive samples must be verified using Lauryl Tryptose Broth and brilliant green lactose bile broth, followed by count adjustment based on these results; and representative non-sheen colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.

[28] A description of KwikCount™ EC may be obtained from Micrology Laboratories LLC.

[29] Approved for the analyses of *E. coli* in freshwater only.

[30] Verification of colonies by incubation of BHI agar at 10 ± 0.5 °C for 48 ± 3 h is optional. As per the Errata to the 23rd Edition of *Standard Methods for the Examination of Water and Wastewater* "Growth on a BHI agar plate incubated at 10 ± 0.5 °C for 48 ± 3 h is further verification that the colony belongs to the genus Enterococcus."

[31] Method 1623.1 includes updated acceptance criteria for IPR, OPR, and MS/MSD and clarifications and revisions based on the use of Method 1623 for years and technical support questions.

[32] 9221 F.2–2014 This procedure allows for simultaneous detection of *E. coli* and thermotolerant coliforms by adding inverted vials to EC–MUG; the inverted vials collect gas produced by thermotolerant coliforms.

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(b) Certain material is incorporated by reference into this section with the approval of the Director of the Federal Register under 5 U.S.C. 552(a) and 1 CFR part 51. All approved material may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue NW, Room 3334, Washington, DC 20004, (Telephone: 202–566–2426) and is available from the sources listed below. It is also available for inspection at National Archives and Records Administration (NARA). For information on the availability of this material at NARA, email *fedreg.legal@nara.gov,* or go to: *www.archives.gov/federal-register/cfr/ibr-locations.html.*

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(8) Office of Water, U.S. Environmental Protection Agency, Washington, DC (US EPA). Available at *https://www.epa.gov/cwa-methods*

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(ix) 1623.1: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. EPA 816–R–12–001. January 2012. US EPA, Table IH, Note 25.

(x) Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality. December 2011. EPA–821–R–09–002. Table IB, Note 69.

(xi) Method 1664, *n*-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated *n*-Hexane Extractable Material (SGT–HEM; Nonpolar Material) by Extraction and Gravimetry.

Revision A, February 1999. EPA–821–R–98–002. Table IB, Notes 38 and 42.

(xii) Method 1664, *n*-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated *n*-Hexane Extractable Material (SGT–HEM; Nonpolar Material) by Extraction and Gravimetry, Revision B, February 2010. EPA–821–R–10–001. Table IB, Notes 38 and 42.

(xiii) Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels. July 1996. Table IB, Note 43.

(xiv) Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using Lauryl Tryptose Broth (LTB) and EC Medium. September 2014. EPA–821–R–14–009.Table IA, Note 15.

(xv) Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A–1 Medium. July 2006. EPA 821–R–06–013. Table IA, Note 20.

(xvi) Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. September 2014. EPA 821–R–14–012. Table IA, Note 23.

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(xiv) 2540, solids. 2015. Table IB.

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(xxxix) 4500-CN^-, Cyanide. 2016. Table IB.

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(xliv) 4500-NO_3^-, Nitrogen (Nitrate). 2016. Table IB.

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(xlvi) 4500-O, Oxygen (Dissolved). 2016. Table IB.

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(lii) 5210, Biochemical Oxygen Demand (BOD). 2016. Table IB.

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(liv) 5310, Total Organic Carbon (TOC). 2014. Table IB.

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(lxvii) 9221 Multiple-Tube Fermentation Technique for Members of the Coliform Group. 2014. Table IA, Notes 12 and 14; Table IH, Notes 10 and 12.

(lxviii) 9222, Membrane Filter Technique for Members of the Coliform Group. 2015. Table IA; Table IH, Note 17.

(lxix) 9223 Enzyme Substrate Coliform Test. 2016. Table IA; Table IH.

(lxx) 9230 Fecal Enterococcus/Streptococcus Groups. 2013. Table IA; Table IH.

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(v) ASTM D511–14, Standard Test Methods for Calcium and Magnesium in Water. November 2014. Table IB.

(vi) ASTM D512–12, Standard Test Methods for Chloride Ion in Water. July 2012. Table IB.

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(viii) ASTM D516–16, Standard Test Method for Sulfate Ion in Water, June 2016. Table IB.

(ix) ASTM D858–17, Standard Test Methods for Manganese in Water. June 2017. Table IB.

(x) ASTM D859–16, Standard Test Method for Silica in Water. June 2016. Table IB.

(xi) ASTM D888–12, Standard Test Methods for Dissolved Oxygen in Water. March 2012. Table IB.

(xii) ASTM D1067–16, Standard Test Methods for Acidity or Alkalinity of Water. June 2016. Table IB.

(xiii) ASTM D1068–15, Standard Test Methods for Iron in Water. October 2015. Table IB.

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(xv) ASTM D1126–17, Standard Test Method for Hardness in Water. December 2017. Table IB.

(xvi) ASTM D1179–16, Standard Test Methods for Fluoride Ion in Water. June 2016. Table IB.

(xvii) ASTM D1246–16, Standard Test Method for Bromide Ion in Water. June 2016. Table IB.

(xviii) ASTM D1252–06 (Reapproved 2012), Standard Test Methods for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Water. June 2012. Table IB.

(xix) ASTM D1253–14, Standard Test Method for Residual Chlorine in Water. February 2014. Table IB.

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(xxi) ASTM D1426–15, Standard Test Methods for Ammonia Nitrogen in Water. April 2015. Table IB.

(xxii) ASTM D1687–17, Standard Test Methods for Chromium in Water. July 2017. Table IB.

(xxiii) ASTM D1688–17, Standard Test Methods for Copper in Water. July 2017. Table IB.

(xxiv) ASTM D1691–17, Standard Test Methods for Zinc in Water. June 2017. Table IB.

(xxv) ASTM D1783–01 (Reapproved 2012), Standard Test Methods for Phenolic Compounds in Water. August 2012. Table IB.

(xxvi) ASTM D1886–14, Standard Test Methods for Nickel in Water. November 2014. Table IB.

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(xxxi) ASTM D2036–09 (Reapproved 2015), Standard Test Methods for Cyanides in Water. July 2015. Table IB.

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(xxxiv) ASTM D2972–15, Standard Tests Method for Arsenic in Water. March 2015. Table IB.

(xxxv) ASTM D3223–17, Standard Test Method for Total Mercury in Water. June 2017. Table IB.

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(xxxvii) ASTM D3373–17, Standard Test Method for Vanadium in Water. June 2017. Table IB.

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(xxxix) ASTM D3557–17, Standard Test Method for Cadmium in Water. June 2017. Table IB.

(xl) ASTM D3558–15, Standard Test Method for Cobalt in Water. March 2015. Table IB.

(xli) ASTM D3559–15, Standard Test Methods for Lead in Water. October 2015. Table IB.

(xlii) ASTM D3590–17, Standard Test Methods for Total Kjeldahl Nitrogen in Water. June 2017. Table IB.

(xliii) ASTM D3645–15, Standard Test Methods for Beryllium in Water. March 2015. Table IB.

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(xlv) ASTM D3859–15, Standard Test Methods for Selenium in Water. April 2015. Table IB.

(xlvi) ASTM D3867–16, Standard Test Method for Nitrite-Nitrate in Water. June 2016. Table IB.

(xlvii) ASTM D4190–15, Standard Test Method for Elements in Water by Direct-Current Plasma Atomic Emission Spectroscopy. March 2015. Table IB.

(xlviii) ASTM D4282–15, Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion. July 2015. Table IB.

(xlix) ASTM D4327–17, Standard Test Method for Anions in Water by Suppressed Ion Chromatography. December 2017. Table IB.

(l) ASTM D4382–18, Standard Test Method for Barium in Water, Atomic Absorption Spectrophotometry, Graphite Furnace. May 2018. Table IB.

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(lii) ASTM D4658–15, Standard Test Method for Sulfide Ion in Water. April 2015. Table IB.

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(liv) ASTM D4839–03 (Reapproved 2017), Standard Test Method for Total Carbon and Organic Carbon in Water by Ultraviolet, or Persulfate Oxidation, or Both, and Infrared Detection. December 2017. Table IB.

(lv) ASTM D5257–17, Standard Test Method for Dissolved Hexavalent Chromium in Water by Ion Chromatography. December 2017. Table IB.

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(lviii) ASTM D5673–16, Standard Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry. February 2016. Table IB.

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(lxi) ASTM. D6508–15, Standard Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. October 2015. Table IB, Note 54.

(lxii) ASTM. D6888–16, Standard Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection. June 2016. Table IB, Note 59.

(lxiii) ASTM. D6919–17, Standard Test Method for Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography. June 2017. Table IB.

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(lxiv) ASTM. D7065–17, Standard Test Method for Determination of Nonylphenol, Bisphenol A, *p-tert*-Octylphenol, Nonylphenol Monoethoxylate and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass Spectrometry. January 2018. Table IC.

(lxv) ASTM. D7237–15a, Standard Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection. June 2015. Table IB.

(lxvi) ASTM. D7284–13 (Reapproved 2017), Standard Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection. July 2017. Table IB.

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(lxviii) ASTM. D7511–12 (Reapproved 2017), Standard Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection. July 2017. Table IB.

(lxix) ASTM. D7573–09 (Reapproved 2017), Standard Test Method for Total Carbon and Organic Carbon in Water by High Temperature Catalytic Combustion and Infrared Detection, February 2017. Table IB.

(lxx) ASTM D7781–14 Standard Test Method for Nitrate-Nitrite in Water by Nitrate Reductase, May 2014. Table IB.

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(19) FIAlab Instruments, Inc., 334 2151 N Northlake Way, Seattle, WA 98103. Telephone: 425–376–0450

(i) Method 100, Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Fluorescence Detector Analysis, April 4, 2018. Table IB, Note 82.

(ii) [Reserved]

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(26) MACHEREY-NAGEL GmbH and Co., 2850 Emrick Blvd., Bethlehem, PA 18020. Telephone: 888–321–6224.

(i) Method 036/038 NANOCOLOR® COD LR/HR, Spectrophotometric Measurement of Chemical Oxygen Demand in Water and Wastewater, Revision 1.5, May, 2018. Table IB, Note 83.

(ii) [Reserved]

(27) Micrology Laboratories, LLC, 1303 Eisenhower Drive, Goshen, IN 46526. Telephone: 574–533–3351.

(i) KwikCount™ EC Medium *E. coli* enzyme substrate test, Rapid Detection of *E. coli* in Beach Water By KwikCount™ EC Membrane Filtration. 2014. Table IH, Notes 28 and 29.

(ii) [Reserved]

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(ii) Determination of Heat Purgeable and Ambient Purgeable Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry. Chapter 12 of Section B, Methods of the National Water Quality Laboratory, of Book 5, Laboratory Analysis. 2016.

(iii) Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, editors, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. Table IB, Note 8.

(iv) 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. 1989. Table IB, Notes 2 and 79.

(v) 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. Table IB, Note 24; Table ID, Note 4.

(vi) OFR 76–177, Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters. 1976. Table IE, Note 2.

(vii) OFR 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. Table ID, Note 14.

(viii) OFR 92–146, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by a Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis. 1992. Table IB, Note 48.

(ix) OFR 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. Table IB, Note 51 and 80; Table IC, Note 9.

(x) OFR 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. Table IB, Note 46.

(xi) OFR 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. Table ID, Note 9.

(xii) OFR 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. Table ID, Note 11.

(xiii) OFR 97–198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1997. Table IB, Note 47.

(xiv) OFR 97–829, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of 86 Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry, Including Detections Less Than Reporting Limits. 1999. Table IC, Note 13.

(xv) OFR 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. Table IB, Notes 50 and 81.

(xvi) OFR 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. Table IB, Note 49.

(xvii) OFR 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. Table IB, Note 45.

(xviii) 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. Table IB, Note 70.

(xix) U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. Table IA, Note 4; Table IH, Note 4.

(xx) Water-Resources Investigation Report 01–4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Moderate-Use Pesticides and Selected Degradates in Water by C–18 Solid-Phase Extraction and Gas Chromatography/Mass Spectrometry. 2001. Table ID, Note 13.

(xxi) 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. Table IB, Note 71.

(xxii) Water-Resources Investigation 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. Table ID, Note 12.

(xxiii) 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. Table IB, Note 32.

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

Parameter No./name	Container [1]	Preservation [2] [3]	Maximum holding time [4]
Table IA—Bacterial Tests			
1–5. Coliform, total, fecal, and *E. coli*.	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	8 hours.[22] [23]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	8 hours.[22]
7. Enterococci	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	8 hours.[22]

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

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
8. *Salmonella*	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$[5]	8 hours.[22]
Table IA—Aquatic Toxicity Tests			
9–12. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C[16]	36 hours.
Table IB—Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6 °C[18]	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C[18]	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C[18]	48 hours.
10. Boron	P, FP, or Quartz	HNO_3 to pH <2	6 months.
11. Bromide	P, FP, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous.	P, FP G	Cool, ≤6 °C[18]	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
16. Chloride	P, FP, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes.
21. Color	P, FP, G	Cool, ≤6 °C[18]	48 hours.
23–24. Cyanide, total or available (or CATC) and free.	P, FP, G	Cool, ≤6 °C[18], NaOH to pH >10[5,6], reducing agent if oxidizer present.	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH <2	6 months.
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
Table IB—Metals[7]			
18. Chromium VI	P, FP, G	Cool, ≤6 °C[18], pH = 9.3 – 9.7[20]	28 days.
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH <2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap[17]	5 mL/L 12N HCl or 5 mL/L BrCl[17]	90 days.[17]
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO_3 to pH <2, or at least 24 hours prior to analysis[19].	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C[18]	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C[18]	48 hours.
41. Oil and grease	G	Cool to ≤6 °C[18], HCl or H_2SO_4 to pH <2.	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤6 °C[18], HCl, H_2SO_4, or H_3PO_4 to pH <2.	28 days.
44. Orthophosphate	P, FP, G	Cool, to ≤6 °C[18,24]	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
49. Phosphorus (elemental)	G	Cool, ≤6 °C[18]	48 hours.
50. Phosphorus, total	P, FP, G	Cool, ≤6 °C[18], H_2SO_4 to pH <2	28 days.
53. Residue, total	P, FP, G	Cool, ≤6 °C[18]	7 days.
54. Residue, Filterable (TDS)	P, FP, G	Cool, ≤6 °C[18]	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C[18]	7 days.
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C[18]	48 hours.
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C[18]	7 days.
61. Silica	P or Quartz	Cool, ≤6 °C[18]	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C[18]	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C[18]	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C[18], add zinc acetate plus sodium hydroxide to pH >9.	7 days.
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes.
68. Surfactants	P, FP, G	Cool, ≤6 °C[18]	48 hours.
69. Temperature	P, FP, G	None required	Analyze within 15 minutes.
73. Turbidity	P, FP, G	Cool, ≤6 °C[18]	48 hours.
Table IC—Organic Tests[8]			
13, 18–20, 22, 24, 25, 27, 28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9].	14 days.[9]

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

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
26. 2-Chloroethylvinyl ether	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons.	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], HCl to pH 2 [9].	14 days.[9]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$, pH to 4–5 [10].	14 days [10]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols [11].	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$	7 days until extraction, 40 days after extraction.
7, 38. Benzidines [11] [12]	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters [11].	G, FP-lined cap	Cool, ≤6 °C [18]	7 days until extraction, 40 days after extraction.
82–84. Nitrosamines [11] [14]	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
88–94. PCBs [11]	G, FP-lined cap	Cool, ≤6 °C [18]	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone [11].	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons [11].	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers [11]	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction.
29, 35–37, 63–65, 107. Chlorinated hydrocarbons [11].	G, FP-lined cap	Cool, ≤6 °C [18]	7 days until extraction, 40 days after extraction.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs [11].	G	See footnote 11	See footnote 11.
Aqueous Samples: Field and Lab Preservation.	G	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], pH <9.	1 year.
Solids and Mixed-Phase Samples: Field Preservation.	G	Cool, ≤6 °C [18]	7 days.
Tissue Samples: Field Preservation.	G	Cool, ≤6 °C [18]	24 hours.
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation.	G	Freeze, ≤ −10 °C	1 year.
114–118. Alkylated phenols	G	Cool, <6 °C, H_2SO_4 to pH <2	28 days until extraction, 40 days after extraction.
119. Adsorbable Organic Halides (AOX).	G	Cool, <6 °C, 0.008% $Na_2S_2O_3$, HNO_3 to pH <2.	Hold *at least* 3 days, but not more than 6 months.
120. Chlorinated Phenolics	G, FP-lined cap	Cool, <6 °C, 0.008% $Na_2S_2O_3$, H_2SO_4 to pH <2.	30 days until acetylation, 30 days after acetylation.
Table ID—Pesticides Tests			
1–70. Pesticides [11]	G, FP-lined cap	Cool, ≤6 °C [18], pH 5–9 [15]	7 days until extraction, 40 days after extraction.
Table IE—Radiological Tests			
1–5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH <2	6 months.
Table IH—Bacterial Tests			
1–4. Coliform, total, fecal	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	8 hours.[22] [23]
5. *E. coli*	PA, G	Cool, <10 °C, 0. 008% $Na_2S_2O_3$ [5]	8 hours.[22]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	8 hours.[22]
7. Enterococci	PA, G	Cool, <10 °C, 0. 008% $Na_2S_2O_3$ [5]	8 hours.[22]
Table IH—Protozoan Tests			
8. *Cryptosporidium*	LDPE; field filtration	1–10 °C	96 hours.[21]
9. *Giardia*	LDPE; field filtration	1–10 °C	96 hours.[21]

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

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[2] Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (*e.g.,* using a 24-hour composite sample; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (*e.g.,* 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.24(f)(14)(iv) and (v) (volatile organics).

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirement of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater; Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory have data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional ATP Coordinator under Sec. 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (*e.g.,* using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; *e.g.,* November 14–15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; *e.g.,* November 14–15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0–6 °C, with minimum head space.

[5] ASTM D7365–09a specifies treatment options for samples containing oxidants (*e.g.,* chlorine) for cyanide analyses. Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (23rd edition) addresses dechlorination procedures for microbiological analyses.

[6] Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365–09a(15). There may be interferences that are not mitigated by the analytical test methods or D7365–09a(15). Any technique for removal or suppression of interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365–09a(15) or the analytical test method must be documented along with supporting data.

[7] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (*e.g.,* using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (*e.g.,* by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

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

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

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

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

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 30 days at <0 °C.

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

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

[16] 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 "≤ °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).

■ 3. Amend § 136.6 by adding paragraph (b)(4)(xxiii) to read as follows:

§ 136.6 Method modifications and analytical requirements.

* * * * *

(b) * * *

(4) * * *

(xxiii) When analyzing metals by inductively coupled plasma-atomic emission spectroscopy, inductively coupled plasma-mass spectrometry, and stabilized temperature graphite furnace atomic absorption, closed-vessel microwave digestion of wastewater samples is allowed as alternative heating source for Method 200.2—''Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements'' for the following elements: Aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, selenium, silver, sodium, thallium, tin, titanium, vanadium, zinc, provided the performance specifications in the relevant determinative method are met. (Note that this list does not include Mercury.) Each laboratory determining total recoverable metals is required to operate a formal quality control (QC) program. The minimum requirements include initial demonstration of capability, method detection limit (MDL), analysis of reagent blanks, fortified blanks, matrix spike samples, and blind proficiency testing samples, as continuing quality control checks on performance. The laboratory is required to maintain performance records on file that define the quality of the data generated.

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