ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[EPA–HQ–OW–2022–0901; FRL–9346–01–OW]

RIN 2040–AG25

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 samples for reporting under EPA's National Pollutant Discharge Elimination System (NPDES) permit program. The Clean Water Act (CWA) requires EPA to promulgate these test procedures (analytical methods) for analysis of pollutants. EPA anticipates that these proposed changes would 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 and make a series of minor changes and corrections to existing approved methods. As such, EPA expects that there would be no negative economic impacts resulting from these proposed changes.

DATES: Comments on this proposed rule must be received on or before April 24, 2023.

ADDRESSES: You may send comments, identified by Docket ID No. EPA–HQ–OW–2022–0901 by any of the following methods:

- *Federal eRulemaking Portal: https://www.regulations.gov* (our preferred method). Follow the online instructions for submitting comments.
- *Email: OW-Docket@epa.gov.* Include Docket ID No. EPA–HQ–OW–2022–0901 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 or 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 the ''Public Participation'' heading of the **SUPPLEMENTARY INFORMATION** section of this document.

FOR FURTHER INFORMATION CONTACT: Tracy Bone, Engineering and Analysis Division (4303T), Office of Water, Environmental Protection Agency, 1200 Pennsylvania Avenue NW, Washington, DC 20460–0001; telephone number: 202–564–5257; email address: *Bone.tracy@epa.gov.*

SUPPLEMENTARY INFORMATION:

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I. Public Participation

A. Written Comments

Submit your comments, identified by Docket ID No. EPA–HQ–OW–2022–0901, at *https://www.regulations.gov* (our preferred method), or the other methods identified in the **ADDRESSES** section. Once submitted, comments cannot be edited or removed from the docket. EPA may publish any comment received to its public docket. Do not submit to EPA's docket at *https://www.regulations.gov* any information you consider to be Confidential Business Information (CBI), Proprietary Business Information (PBI), or other information whose disclosure is restricted by statute. 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. 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). Please visit *https://www.epa.gov/dockets/commenting-epa-dockets* for additional submission methods; the full EPA public comment policy; information about CBI, PBI, or multimedia submissions; and general guidance on making effective comments. Publicly available docket materials are available electronically in *www.regulations.gov* 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. Any copyright material can be viewed at the Reading Room, please contact the EPA Docket Center, public Reading Room. The telephone number for the Public Reading Room is 202–566–1744, and the telephone number for the Water Docket is 202–566–2426. Fax: 202–566–9744. Email: *docket-customerservice@epa.gov.*

II. 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 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; the environmental monitoring industry.
Municipalities	Publicly Owned Treatment Works (POTWs) or other municipality-owned facilities that must conduct monitoring to comply with NPDES permits.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be affected by this action. This table lists types of entities that 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 action is the Agency taking?

Periodically, 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 category is updated versions of EPA methods currently approved in 40 CFR part 136. The second category is new or revised methods published by a voluntary consensus standard body (VCSB) or the United States Geologic Survey (USGS) that are similar to methods previously adopted as EPA-approved methods in 40 CFR part 136. The third category is methods EPA has reviewed under the agency's national Alternate Test Procedure (ATP) program and preliminarily concluded are appropriate for nationwide use. Finally, EPA is proposing certain corrections or amendments to the text and tables of 40 CFR part 136. 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.

C. What is the agency's authority for taking this action?

EPA is proposing this regulation under the authorities of sections 301(a), 304(h), and 501(a) of the CWA; 33 U.S.C. 1251, 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 EPA Administrator 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].'' 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).

III. Background

This preamble describes the abbreviations and acronyms; reasons for the proposed rule; and a summary of the proposed changes and clarifications; the legal authority for the proposed rule; methods incorporated by reference; a summary of the proposed changes and clarifications and solicits comment from the public.

Abbreviations and Acronyms Used in the Preamble and Proposed Rule Text

ADMI: American Dye Manufacturers Institute
ASTM: ASTM International[1]
ATP: Alternate Test Procedure
BHI: Brain heart infusion
BOD_5: 5-day Biochemical Oxygen Demand
CATC: Cyanide Amenable to Chlorination
CBI: Confidential Business Information
CFR: Code of Federal Regulations
CIE: Capillary Ion Electrophoresis
CNCl: Cyanogen Chloride
CWA: Clean Water Act
EC–MUG: EC broth with 4-methylumbelliferyl-β-D-glucuronide
EDTA: Ethylenediaminetetraacetic acid
EPA: Environmental Protection Agency
DO: Dissolved Oxygen
GC: Gas Chromatography
GC/MS/MS: Gas Chromatography-Tandem Mass Spectrometry
GC/HRMS: Gas Chromatography-High Resolution Mass Spectrometry
ICP/AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy
MIBK: Methyl Isobutyl Ketone
NED: *N*-(1-naphthyl)-ethylenediamine dihydrochloride
MF: Membrane Filtration
$MgCl_2$: Magnesium Chloride
MPN: Most Probable Number
nm: Nanometer
NPDES: National Pollutant Discharge Elimination System
NTTAA: National Technology Transfer and Advancement Act
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

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 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 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, 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 category is updated versions of EPA methods currently approved in 40 CFR part 136. The second 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 third category is methods EPA has reviewed under the Agency's national ATP program and preliminarily concluded are appropriate for nationwide use. Finally, EPA is proposing certain corrections or amendments to the text and tables of 40 CFR part 136. 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

EPA proposes to approve revised versions of the EPA membrane filtration methods 1103.2, 1106.2, 1600.1, and 1603.1 found in Tables IA and IH. These methods were approved from 2002 to 2014. The revisions include standardizing language between the related methods, updating to reflect current lab practices and clarifying edits. Copies of these proposed method updated versions are available in the docket to this rule.

These methods each describe a membrane filter (MF) procedure for the detection and enumeration of either enterococci or *Escherichia coli* bacteria

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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).

by their growth after incubation on selective media. These methods provide a direct count of bacteria in water samples based on the development of colonies on the surface of the membrane filter.

1. *E. coli.* Method 1103.2 describes a MF procedure for the detection and enumeration of Escherichia coli bacteria in ambient (fresh) water and is currently approved in Table IH. This is a two-step method which requires transferring the membrane filter after incubation on membrane-Thermotolerant Escherichia coli Agar (mTEC) to a pad saturated with urea substrate.

2. *Enterococci.* Method 1106.2 describes a MF procedure for the detection and enumeration of enterococci bacteria in ambient water and is currently approved in Table IH. This is a two-step method which requires transferring the membrane filter after incubation on membraneEnterococcus (mE) agar to Esculin Iron Agar (EIA) medium.

3. *Enterococci.* Method 1600.1 describes a MF procedure for the detection and enumeration of enterococci bacteria in ambient (fresh and marine) water and wastewater and is currently approved in Tables IA and IH. This is a single-step method that is a modification of EPA Method 1106.1 (mE–EIA). The membrane filter containing the bacterial cells is placed on membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI).

4. *E. coli.* Method 1603.1 describes a MF procedure for the detection and enumeration of thermotolerant Escherichia coli bacteria in ambient (fresh) waters and wastewaters using Modified membrane-Thermotolerant Escherichia coli Agar (modified mTEC) and is currently approved in Table IA and IH.

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

EPA is proposing to approve new versions of ASTM methods previously approved in 40 CFR part 136. These changes to currently approved ASTM methods in 40 CFR part 136 include minor clarifications and editorial changes. As an example, ASTM added text to the appropriate method scope sections to indicate that the method was developed in accordance with the ‘‘Decision on Principles for the Development of International Standards, Guides and Recommendations’’ issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee. None of these proposed changes will affect the performance of the method. The following describes the changes to current ASTM methods that EPA proposes to include in 40 CFR part 136. Each entry contains (in the following order): the parameter, proposed ASTM method number (the last two digits in the method number represent the year ASTM published), a brief description of the analytical technique, and a brief description of any minor procedural changes (if there are any) in this revision from the last approved version of the method. Method revisions that are only formatting in nature will have no description of the changes. The methods listed below are organized according to the table at 40 CFR part 136 in the order in which they appear.

EPA proposes the following changes to ASTM methods found in Table IB, and Table II at 40 CFR part 136:

1. *Dissolved Oxygen.* D888–18 (A, B, C), Dissolved Oxygen, Winkler, Electrode, Luminescent-based Sensor. Standard D888–18A 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. Standard D888–18B 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. Standard D888–18C measures dissolved oxygen with a luminescence-based sensor probe that employs frequency domain lifetime-based luminescence quenching and signal processing. The 2012 versions, D888–12 (A), (B) and (C), currently are approved in Table IB for determination of dissolved oxygen.

2. *Hydrogen Ion (pH).* In D1293–18 (A, B), pH, Electrometric. The activity of hydrogen ion (H+) in the sample is determined electrometrically with an ion-selective electrode in comparison to at least two standard reference buffers and pH is reported as the negative log of that activity. The 1999 version currently is approved in Table IB.

3. *Metals Series.* In D1976–20, Elements in Water by Inductively-Coupled Plasma Atomic Emission Spectroscopy for determination of aluminum, antimony, arsenic, beryllium, boron, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, selenium, silver, thallium, vanadium, and zinc. The sample is acid digested and analyzed by inductively-coupled plasma atomic emission spectroscopy (ICP/AES) for the simultaneous or sequential determination of 29 elements. The changes include changing the initial instrument calibration from using four standards as the first option to using only one standard and a calibration blank. The 2012 version of this method, D1976–12, currently is approved in Table IB for 20 of the 29 elements.

4. *Surfactants.* In D2330–20, Methylene Blue Active Substances, the sample is mixed with an acidic aqueous solution of methylene blue reagent, which forms a blue-colored ion pair with any anionic surfactants which is subsequently extracted with chloroform and washed with an acidic solution to remove interferences. The intensity of the blue color is measured using a photometer at 650 nanometers (nm). The concentration of methylene blue active substances is determined in comparison to a standard curve. The 2002 version, D2330–02, currently is approved in Table IB for determination of surfactants.

5. *Residue, filterable and nonfilterable.* In D5907–18 (A and B), Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) under Test Method A, an aliquot of the sample is filtered through a glass fiber filter and the solids trapped on the filter are dried at 105 °C and weighed to determine the nonfilterable material (total suspended solids) by difference. Under Test Method B, the filtrate from Test Method A, or a separate filtrate, is evaporated to dryness at 180 °C and the residue weighed to determine the total dissolved solids. The 2013 version is currently approved in Table IB.

6. *Cyanide—Free.* In D7237–18, 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. This version also added new information about sulfide interferences and potential mitigation strategies that EPA anticipates will improve data quality. There are no other procedural changes. The 2015 version, D7237–15, currently is approved in Table IB for determination of free cyanide.

7. *Cyanide—Total.* In D7284–20, 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. The 2017 reapproval of D7284–13 currently is approved in Table IB for determination of total cyanide.

8. *Organic Carbon.* In D7573–18a^{e1}, Total Organic Carbon, Combustion, the sample is sparged with an inert gas to remove dissolved inorganic carbon, acidified, and then combusted at high temperature to convert organic carbon to carbon dioxide. The carbon dioxide is measured with an infra-red detector. This version also adds data from an interlaboratory method validation study and new method detection limit values, but there are no procedural changes. The 2017 reapproval of D7573–09 currently is approved in Table IB for determination of total organic carbon (TOC).

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

EPA is proposing to approve new versions of methods developed by the Standard Methods Committee that were previously approved in 40 CFR part 136. Standard Methods has reviewed many of their methods in preparation for releasing the next edition of ''Standard Methods for the Examination of Water & Wastewater.'' The newer versions provide clarifications and make editorial corrections. These edits include removal of referents to specific brand names and trademarks, incorporation of footnotes into the text, a reformatting of figures, tables and reference lists, removal of bibliographical references that are no longer available, small editorial changes based on current style guides and changes to scientific publishing standards, and minor clarifications to procedures based on input from users. For example, the revisions replace distilled water with reagent water in all methods. As was the case with the previous methods update rule (86 FR 27226, May 19, 2021), 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. 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.,* 3111 C–2019). 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'' that includes a method with the same method number and year of publication.

The proposed revisions to methods previously approved in 40 CFR part 136 will not affect the performance of the method. Below is a list of the methods 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.

EPA proposes to make the following changes to Tables IA, IB, IC, ID and IH at 40 CFR part 136 for the following parameters:

1. *Color.* 2120 B–2021, Visual Comparison Method, is a platinum-cobalt method of measuring color, the unit of color being that produced by one mg platinum per liter in the form of the chloroplatinate ion. The 1:2 ratio of cobalt to platinum resulting from the preparation of the standard platinum-cobalt solution matches the color of natural waters. The 2011 editorial revision currently is approved in Table IB for determination of color. 2120 F–2021, American Dye Manufacturers Institute (ADMI) Weighted-Ordinate Spectrophotometric Method. In accordance with the Adams-Nickerson chromatic value formula, this method calculates single-number color difference values (*i.e.,* uniform color differences). Values are independent of chroma and hue. Transmittance of light is measured spectrophotometrically at multiple wavelengths and converted to a set of abstract numbers, which then are converted to a single number that indicates color value. This number is expressed on a scale used by the ADMI. The 2011 editorial revision currently is approved in Table IB for determination of color.

2. *Turbidity.* 2130 B–2020, Nephelometric Method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the intensity of scattered light, the higher the turbidity. Formazin polymer is used as the primary standard reference suspension. The 2011 editorial revision currently is approved in Table IB for determination of turbidity.

3. *Acidity.* 2310 B–2020, Titration Method measures the hydrogen ions present in a sample as a result of dissociation or hydrolysis of solutes that react with additions of standard alkali. Acidity thus depends on the endpoint pH or indicator used. The construction of a titration curve by recording a sample's pH after successive small, measured additions of titrant permits identification of inflection points and buffering capacity, if any, and allows the acidity to be determined with respect to any pH of interest. Samples of industrial wastes, acid mine drainage, or other solutions that contain appreciable amounts of hydrolyzable metal ions such as iron, aluminum, or manganese are treated with hydrogen peroxide to ensure the oxidation of any reduced forms of polyvalent cations and are boiled to hasten hydrolysis. Acidity results may be highly variable if this procedure is not followed exactly. The 2011 editorial revision currently is approved in Table IB for determination of acidity.

4. *Alkalinity.* 2320 B–2021 Titration Method, measures the hydroxyl ions present in a sample resulting from dissociation or hydrolysis of solutes that react with additions of standard acid. Alkalinity thus depends on the endpoint pH used. For samples of low alkalinity (less than 20 mg/L $CaCO_3$) an extrapolation technique based on the near proportionality of concentration of hydrogen ions to excess of titrant beyond the equivalence point is used. The amount of standard acid required to reduce the pH exactly 0.30 pH unit is measured carefully. Because this change in pH corresponds to an exact doubling of the hydrogen ion concentration, a simple extrapolation can be made to the equivalence point. The 2011 editorial revision currently is approved in Table IB for determination of alkalinity.

5. *Hardness.* 2340 B–2021, Hardness by Calculation is the preferred method for determining hardness by calculating it from the results of separate determinations of calcium and magnesium by any approved method provided that the sum of the lowest point of quantitation for Ca and Mg is below the NPDES permit requirement for hardness. The 2011 editorial revision currently is approved in Table IB for determination of hardness. In 2340 C–2021, Ethylenediaminetetraacetic acid (EDTA) Titrimetric Method, EDTA forms a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as eriochrome black T or calmagite is added to an aqueous solution containing calcium and magnesium ions at a pH of 10.0 ± 0.1, the color of the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the magnesium and calcium has been complexed, the solution turns from wine red to blue, marking the endpoint of the titration. The volume of titrant used is proportional to hardness in the

sample. Magnesium ion must be present to yield a satisfactory endpoint. To ensure this, a small amount of complexometrically neutral magnesium salt of EDTA is added to the buffer; this automatically introduces sufficient magnesium and obviates the need for a blank correction. The 2011 editorial revision currently is approved in Table IB for determination of hardness.

6. *Specific Conductance.* 2510 B–2021 measures conductance (or resistance) in the laboratory using a standard potassium chloride solution and from the corresponding conductivity, a cell constant is calculated. Most conductivity meters do not display the actual solution conductance, or resistance, rather, they generally have a dial that permits the user to adjust the internal cell constant to match the conductivity of a standard. Once the cell constant has been determined, or set, the conductivity of an unknown solution is displayed by the meter. The 2011 editorial revision currently is approved in Table IB for determination of specific conductance.

7. *Residue—Total.* In 2540 B–2020 an aliquot of a well-mixed sample is evaporated in a pre-weighed evaporating dish at 103–105 °C to constant weight in a 103 to 105 °C oven. The increase compared to the empty pre-weighed dish weight represents total solids. The 2015 version of the method currently is approved in Table IB for determination of total residue. In 2540 C–2020, Total Dissolved Solids Dried at 180 °C (Residue—filterable in Table IB) a measured volume of a well-mixed sample is filtered through a glass fiber filter with applied vacuum. The entire exposed surface of the filter is washed with at least 3 successive volumes of reagent-grade water with continued suction until all traces of water are removed. The total filtrate (with washings) is then transferred to a pre-weighed dish and evaporated to dryness. Successive volumes of sample are added to the same dish after evaporation if necessary to yield between 2.5 and 200 mg of dried residue. The evaporated residue is then dried for one hour or more in an oven at 180 °C, cooled in a desiccator to ambient temperature, and weighed until the weight change is less than 0.5 mg. The 2015 version of the method currently is approved in Table IB for determination of filterable residue. In 2540 D–2020, Total Suspended Solids Dried from 103 to 105 °C (Residue—non-filterable total suspended solids (TSS) in Table IB) a well-mixed sample is filtered through a pre-weighed standard glass-fiber filter. The filter and the retained residue are then dried to a constant weight in a 103 to 105 °C oven. The increase in filter weight represents TSS. The 2015 version of the method currently is approved in Table IB for determination of non-filterable residue. In 2540 E–2020, Fixed and Volatile Solids Ignited at 550 °C (Residue—volatile in Table IB) the residue obtained from the determination of total (Method 2540 B), filterable (Method 2540 C), or non-filterable residue (Method 2540 D) is ignited at 550 ± 50 °C in a muffle furnace, cooled in a desiccator to ambient temperature and weighed. Repeated successive cycles of drying, cooling, desiccating, and weighing are performed until the weight change is less than 0.5 mg. The remaining solids are *fixed* total, dissolved, or suspended solids, while those lost to ignition are *volatile* total, dissolved, or suspended solids. The 2015 version of the method currently is approved in Table IB for determination of volatile residue. In 2540 F–2020, Settleable Solids (aka, Residue—settleable in Table IB), a well-mixed sample is used to fill an Imhoff cone or graduated cylinder to the 1–L mark. The sample is allowed to settle for 45 minutes, then gently agitated near the sides of the cone (or graduated cylinder) with a rod or by spinning. The sample is then allowed to settle for another 15 minutes and the volume of settleable solids in the cone (or graduated cylinder) is recorded as mL/L. When applicable, the recorded volume is corrected for interference from pockets of liquid volume. The 2015 version of the method currently is approved in Table IB for determination of settleable residue.

8. *Multiple metals by flame atomic absorption spectrometry.*

a. *3111 B–2019, Direct Air-Acetylene Flame Method.* The 2011 editorial revision currently is approved in Table IB for determination of antimony, cadmium, calcium, chromium, cobalt, copper, gold, iridium, iron, lead, magnesium, manganese, nickel, palladium, platinum, potassium, rhodium, ruthenium, silver, sodium, thallium, tin, and zinc. A sample is aspirated into a flame and the metals are atomized. A light beam is directed through the flame, into a monochromator, and onto a detector that measures the amount of light absorbed by the atomized metal in the flame. Because each metal has its own characteristic absorption wavelength, a source lamp composed of that element is used. The amount of energy at the characteristic wavelength absorbed in the flame is proportional to the concentration of the element in the sample over a limited concentration range.

b. 3111 C–2019, Extraction and Air-Acetylene Flame Method consists of chelation with ammonium pyrrolidine dithiocarbamate (APDC) and extraction into methyl isobutyl ketone (MIBK), followed by aspiration into an air-acetylene flame and is suitable for the determination of low concentrations of cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, silver, and zinc. The 2011 editorial revision currently is approved in Table IB for determination of cadmium, chromium, cobalt, copper, iron, lead, nickel, silver, and zinc.

EPA proposes to approve method 3111 C for manganese. This parameter was inadvertently left off in an earlier rulemaking approving method 3111 C.

c. *3111 D–2019, Direct Nitrous Oxide-Acetylene Flame Method.* A sample is aspirated into a flame produced using a mixture of nitrous oxide and acetylene and the metals are atomized. A light beam is directed through the flame, into a monochromator, and onto a detector that measures the amount of light absorbed by the atomized metal in the flame. The 2011 editorial revision currently is approved in Table IB for determination of aluminum, barium, beryllium, molybdenum, osmium, titanium, and vanadium. In addition, EPA proposes to approve method 3111 D for calcium. This parameter was inadvertently left off in an earlier rulemaking approving method 3111 D.

d. *3111 E–2019, Extraction and Nitrous Oxide-Acetylene Flame Method.* The method consists of chelation with 8-hydroxyquinoline, extraction with MIBK, and aspiration into a nitrous oxide-acetylene flame and is suitable for the determination of aluminum at concentrations less than 900 µg/L and beryllium at concentrations less than 30 µg/L. The 2011 editorial revision currently is approved in Table IB for determination of aluminum, and beryllium.

9. *Mercury—Total.* 3112 B–2020, Metals by Cold-Vapor Atomic Absorption Spectrometric Method is a flameless AA procedure based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury in a sample is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. The 2011 editorial revision currently is approved in Table IB for determination of mercury.

10. *Metals by AA Furnace.* In 3113 B–2020, Electrothermal Atomic Absorption Spectrometric Method, a discrete sample volume is dispensed into the graphite sample tube (or cup). Typically, determinations are made by heating the sample in three or more stages. First, a low current heats the tube to dry the sample. The second, or charring, stage destroys organic matter and volatilizes other matrix components at an intermediate temperature. Finally, a high current heats the tube to incandescence and, in an inert atmosphere, atomizes the element being determined. Additional stages frequently are added to aid in drying and charring, and to clean and cool the tube between samples. The resultant ground-state atomic vapor absorbs monochromatic radiation from the source. A photoelectric detector measures the intensity of transmitted radiation. The inverse of the transmittance is related logarithmically to the absorbance, which is directly proportional to the number density of vaporized ground-state atoms (the Beer-Lambert law) over a limited concentration range. The 2010 version of the method currently is approved in Table IB for determination of aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, nickel, selenium, silver, and tin. Although not specifically listed as target analytes in 3113 B, the 2010 version of the method is also approved in Table IB for determination of gold, thallium, and vanadium, as these elements may also be determined using the method.

11. *Arsenic and Selenium by AA Gaseous Hydride.* 3114 B–2020, Manual Hydride Generation/Atomic Absorption Spectrometric Method is a manual hydride generation method that is applicable to the determination of arsenic and selenium by conversion to their hydrides by sodium borohydride reagent and transport into an atomic absorption atomizer. The 2011 editorial revision currently is approved in Table IB for determination of arsenic and selenium. 3114 C–2020, Continuous Hydride Generation/Atomic Absorption Spectrometric Method is a continuous-flow hydride generation method that is applicable to the determination of arsenic and selenium by conversion to their hydrides by sodium borohydride reagent and transport into an atomic absorption atomizer. The continuous hydride generator offers the advantages of simplicity in operation, excellent reproducibility, low detection limits, and high sample volume throughput for selenium analysis following preparations as described in 3500–Se B or 3114 B.4c and d. The 2011 editorial revision currently is approved in Table IB for determination of arsenic and selenium.

12. *Multiple Metals by ICP/AES (Plasma Emission Spectroscopy).* In 3120 B–2020, an Inductively Coupled Plasma (ICP) source consists of a flowing stream of argon gas ionized by an applied radio frequency field typically oscillating at 27.1 MHz. This field is inductively coupled to the ionized gas by a water-cooled coil surrounding a quartz torch that supports and confines the plasma. A sample aerosol is generated in an appropriate nebulizer and spray chamber and is carried into the plasma through an injector tube located within the torch. The sample aerosol is injected directly into the ICP, subjecting the constituent atoms to temperatures of about 6000 to 8000 °K. Because this results in almost complete dissociation of molecules, significant reduction in chemical interferences is achieved. The high temperature of the plasma excites atomic emission efficiently. Ionization of a high percentage of atoms produces ionic emission spectra. The ICP provides an optically thin source that is not subject to self-absorption except at very high concentrations. Total metals are determined after appropriate digestion. The 2011 editorial revision currently is approved in Table IB for determination of aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, selenium, silica, silver, sodium, thallium, vanadium, and zinc. Although not specifically listed as a target analyte in method 3120 B, the 2011 version of the method is also approved in Table IB for determination of phosphorus because this element may also be determined using the method.

13. *Multiple Metals by Inductively Coupled Plasma-Mass Spectrometry.* In this method, 3125 B–2020, Inductively Coupled Plasma-Mass Spectrometry (ICP–MS) Method, a sample is introduced into an argon-based, high-temperature radio-frequency plasma, usually via pneumatic nebulization. As energy transfers from the plasma to the sample stream, the target element desolvation, atomization, and ionization. The resulting ions are extracted from the plasma through a differential vacuum interface and separated based on their mass-to-charge (m/z) ratio by a mass spectrometer. Typically, either a quadrupole (with or without collision cell technology or dynamic reaction cell) or magnetic sector (high-resolution) mass spectrometer is used. An electron multiplier detector counts the separated ions, and a computer-based data-management system processes the resulting information. The 2011 editorial revision currently is approved in Table IB for determination of aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, potassium, selenium, silver, thallium, vanadium, and zinc. Although not specifically listed as a target analyte in method 3125 B, the 2011 version of the method is also approved in Table IB for determination of boron, calcium, gold, iridium, iron, magnesium, palladium, platinum, potassium, rhodium, ruthenium, silica, sodium, tin, and titanium as these elements may also be determined using the method.

14. *3500 Colorimetric Series for Multiple Metals.*

a. *Aluminum.* In 3500–Al B–2020, Eriochrome Cyanine R Method with Eriochrome cyanine R dye, dilute aluminum solutions buffered to a pH of 6.0 produce a red to pink complex that exhibits maximum absorption at 535 nm. The intensity of the developed color is influenced by the aluminum concentration, reaction time, temperature, pH, alkalinity, and concentration of other ions in the sample. To compensate for color and turbidity, the aluminum in one portion of a sample is complexed with EDTA to provide a blank. The interference of iron and manganese, two elements commonly found in water when aluminum is present, is eliminated by adding ascorbic acid. The 2011 editorial revision currently is approved in Table IB for determination of aluminum.

b. *Arsenic.* In 3500–As B–2020, Silver Diethyldithiocarbamate Method, arsenite, containing trivalent arsenic, is reduced selectively by aqueous sodium borohydride solution to arsine, AsH_3, in an aqueous medium of pH 6. Arsenate, methylarsonic acid, and dimethylarsinic acid are not reduced under these conditions. The generated arsine is swept by a stream of oxygen-free nitrogen from the reduction vessel through a scrubber containing glass wool or cotton impregnated with lead acetate solution into an absorber tube containing silver diethyldithiocarbamate and morpholine dissolved in chloroform. The intensity of the red color that develops is measured at 520 nm. The 2011 editorial revision currently is approved in Table IB for determination of arsenic.

c. *Calcium.* In 3500–Ca B–2020, EDTA Titrimetric Method, EDTA is added to water containing both calcium and magnesium, where it combines first with the calcium. Calcium can be determined directly, with EDTA, when the pH is made sufficiently high that the magnesium is largely precipitated as the hydroxide and an indicator is used that combines with calcium only. Several indicators give a color change when all the calcium has been complexed by the EDTA at a pH of 12 to 13. The 2011 editorial revision currently is approved in Table IB for determination calcium.

d. *Chromium.* 3500–Cr B–2020, Colorimetric Method. This procedure measures only hexavalent chromium, (chromium VI). The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet colored complex of unknown composition is produced. The 2011 editorial revision currently is approved in Table IB for determination of dissolved hexavalent chromium (chromium VI). 3500–Cr C–2020, Ion Chromatographic Method. This method is applicable to determination of dissolved hexavalent chromium in drinking water, groundwater, and industrial wastewater effluents. An aqueous sample is filtered, and its pH adjusted to between 9 and 9.5 with a concentrated buffer. This pH adjustment reduces the solubility of trivalent chromium and preserves the hexavalent chromium oxidation state. The sample is introduced into the instrument's eluent stream of ammonium sulfate and ammonium hydroxide. Trivalent chromium in solution is separated from the hexavalent chromium by the column. After separation, hexavalent chromium reacts with an azide dye to produce a chromogen that is measured at 530 or 540 nm. Hexavalent chromium is identified based on retention time. The 2011 editorial revision currently is approved in Table IB for determination of dissolved hexavalent chromium (chromium VI).

e. *Copper Colorimetric.* In 3500–Cu B–2020, Neocuproine Method, the sample is treated with hydroxylamine hydrochloride to reduce any cupric ions (Cu^{2+}) to cuprous ions (Cu^{+}). Sodium citrate is used to complex metallic ions that might precipitate when the pH is raised. The pH is adjusted to between 4 and 6 with ammonium hydroxide (NH_4OH), a solution of neocuproine (2,9-dimethyl-1,10-phenanthroline) in methanol is added, and the resultant complex is extracted into chloroform ($CHCl_3$). After dilution of the $CHCl_3$ to an exact volume with methanol (CH_3OH), the absorbance of the solution is measured at 457 nm. The 2011 editorial revision currently is approved in Table IB for determination of copper. In 3500–Cu C–2020, Bathocuproine Method, cuprous ion forms a water-soluble orange-colored chelate with disodium bathocuproine disulfonate (sodium 4,4′-(2,9-dimethyl-1,10-phenanthroline-4,7-diyl)dibenzenesulfonate). While the color forms over the pH range 3.5 to 11.0, the recommended pH range is between 4 and 5. The sample is buffered at a pH of about 4.3 and reduced with hydroxylamine hydrochloride. The absorbance is measured at 484 nm. The 2011 editorial revision currently is approved in Table IB for determination of copper.

f. *Potassium.* In 3500–K B–2020, Flame Photometric Method, trace amounts of potassium can be determined in either a direct-reading or internal-standard type of flame photometer at a wavelength of 766.5 nm. The 2011 editorial revision currently is approved in Table IB for determination of potassium. In 3500–K C–2020, Potassium-Selective Electrode Method, potassium ions are measured potentiometrically by using a potassium ion-selective electrode and a double-junction, sleeve-type reference electrode. The analysis is performed with either a pH meter having an expanded millivolt scale capable of being read to the nearest 0.1 mV or a specific-ion meter having a direct concentration scale for potassium. Before measurement, an ionic strength adjustor reagent is added to both standards and samples to maintain a constant ionic strength. The electrode response is measured in standard solutions with potassium concentrations spanning the range of interest using a calibration line derived either by the instrument meter or manually. The electrode response in sample solutions is measured following the same procedure and potassium concentration determined from the calibration line or instrument direct readout. The 2011 editorial revision currently is approved in Table IB for determination of potassium.

g. *Manganese.* In 3500–Mn B–2020, Persulfate Method, persulfate oxidation of soluble manganous compounds to form permanganate is carried out in the presence of silver nitrate. The resulting color is stable for at least 24 hours if excess persulfate is present and organic matter is absent. The 2011 editorial revision currently is approved in Table IB for determination of manganese.

h. *Sodium.* In 3500–Na B–2020, Flame Emission Photometric Method a sample is nebulized into a gas flame under carefully controlled, reproducible excitation conditions. The sodium resonant spectral line at 589 nm is isolated by interference filters or by light-dispersing devices such as prisms or gratings. Emission light intensity is measured by a phototube, photomultiplier, or photodiode. The light intensity at 589 nm is approximately proportional to the sodium concentration. The 2011 editorial revision currently is approved in Table IB for determination of sodium.

i. *Lead.* In 3500–Pb B–2020, Dithizone Method, an acidified sample containing microgram quantities of lead is mixed with ammoniacal citrate-cyanide reducing solution and extracted with dithizone in chloroform ($CHCl_3$) to form a cherry-red lead dithizonate. The color of the mixed color solution is measured photometrically. The 2011 editorial revision currently is approved in Table IB for determination of lead.

j. *Zinc.* 3500–Zn B–2020, Zincon Method. Zinc forms a blue complex with zincon (2-carboxy-2′-hydroxy-5′-sulfoformazyl benzene) in a solution buffered to pH 9.0. Other heavy metals likewise form colored complexes with zincon. Cyanide is added to complex zinc and heavy metals. Cyclohexanone is added to selectively free zinc from its cyanide complex so that it can be complexed with zincon to form a blue color which is measured spectrophotometrically at 620 nm. Sodium ascorbate reduces manganese interference. The developed color is stable except in the presence of copper. The 2011 editorial revision currently is approved in Table IB for determination of zinc.

15. *4110 Series, Ion Chromatography.*

a. In 4110 B–2020, Ion Chromatography with Chemical Suppression of Eluent Conductivity, is approved in Table IB for determination of bromide, chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate. A water sample is injected into a stream of eluent and passed through a series of ion exchangers. The anions of interest are separated based on their relative affinities for a low-capacity, strongly basic anion exchanger (guard and analytical columns). The separated anions are directed through a suppressor device that provides continuous suppression of eluent conductivity and enhances analyte response. In the suppressor, the separated anions are converted to their highly conductive acid forms while the conductivity of the eluent is greatly decreased. The separated anions in their acid forms are measured by conductivity. They are identified based on retention time as compared to standards. Quantitation is by measurement of peak area or peak height. The 2011 editorial revision currently is approved in Table IB for determination of bromide, chloride, fluoride, nitrate, combined nitrate-nitrite, nitrite, orthophosphate, and sulfate.

b. *4110 C–2020, Single-Column Ion Chromatography with Direct Conductivity Detection.* An aqueous sample is injected into an ion chromatograph consisting of an injector port, analytical column, and conductivity detector. The sample merges with the eluent stream and is pumped through the analytical column where the anions are separated based on their affinity for the active sites of the column packing material. Concentrations are determined by direct conductivity detection without chemical suppression. The 2011 editorial revision currently is approved in Table IB for determination of bromide, chloride, fluoride, nitrate, combined nitrate-nitrite, nitrite, orthophosphate, and sulfate.

c. *4110 D–2020, Ion Chromatographic Determination of Oxyhalides and Bromide.* The sample is analyzed in a manner similar to that in 4110 B–2020. However, bromate has been shown to be subject to positive interferences in some matrices. The interference is noticeable usually as a flattened peak. It often can be eliminated by passing the sample through an H^+ off-line solid-phase extraction (SPE) cartridge, by selection of a different column-eluent combination, or by diluting the eluent, which will increase retention times and spread the chromatogram. Additionally, chloride or a nontarget analyte present in unusually high concentration may overlap with a target analyte sufficiently to cause problems in quantitation or may cause retention-time shifts. Dilution of the sample may resolve this problem. The 2011 editorial revision currently is approved in Table IB for determination of bromide.

16. *Inorganic Anions by CIE/UV (Capillary Ion Electrophoresis).* In 4140 B–2020, Capillary Ion Electrophoresis with Indirect UV Detection, the sample is introduced at the cathodic end of the capillary and anions are separated based on their differences in mobility in the electric field as they migrate through the capillary. Cations migrate in the opposite direction and are not detected. Water and neutral organics are not attracted toward the anode. They migrate after the anions and thus do not interfere with anion analysis. Anions are detected as they displace charge-for-charge the UV-absorbing electrolyte anion (chromate), causing a net decrease in UV absorbance in the analyte anion zone compared to the background electrolyte. Detector polarity is reversed to provide positive millivolt response to the data system. As in chromatography, the analytes are identified by their migration time and quantitated by using time-corrected peak area relative to standards. The 2011 editorial revision currently is approved in Table IB for determination of bromide, chloride, fluoride, nitrate, combined nitrate-nitrite, nitrite, orthophosphate, and sulfate.

17. *4500 Series, Chloride.*

a. *4500–Cl^- B–2021, Titrimetric Method.* In a neutral or slightly alkaline solution, potassium chromate can indicate the endpoint of the silver nitrate titration of chloride. Silver chloride is precipitated quantitatively before red silver chromate is formed. In this version of the method approved by the Standard Methods Committee in 2021, additional information regarding removal of interferences caused by sulfide, thiosulfate, and sulfite ions by digestion of the sample with hydrogen peroxide prior to titration has been added to the sample preparation procedures. A tighter pH range of 8–10, as opposed to 7–10, is specified for adjustment of the pH of the sample prior to titration. A reference has been added for the 2021 Standard Methods Joint Task Group validation report titled: ‘‘Interlaboratory validation study for the use of H_2O_2 with boiling for determining Cl^-.’’ The 2011 editorial revision currently is approved in Table IB for determination of chloride.

b. *4500–Cl^- C–2021, Mercuric Nitrate Method.* Chloride can be titrated with mercuric nitrate, $Hg(NO_3)_2$, because of the formation of soluble, slightly dissociated mercuric chloride. In the pH range 2.3 to 2.8, diphenylcarbazone indicates the titration endpoint by formation of a purple complex with the excess mercuric ions. Xylene cyanol FF serves as a pH indicator and endpoint enhancer. Increasing the strength of the titrant and modifying the indicator mixtures extend the range of measurable chloride concentrations. The 2011 editorial revision currently is approved in Table IB for determination of chloride.

c. *4500–Cl^- D–2021, Potentiometric Method.* Chloride is determined by potentiometric titration with silver nitrate solution with a glass and silver-silver chloride electrode system. During titration, an electronic voltmeter is used to detect the change in potential between the two electrodes. The endpoint of the titration is that instrument reading at which the greatest change in voltage has occurred for a small and constant increment of silver nitrate added. The 2011 editorial revision currently is approved in Table IB for determination of chloride.

d. *4500–Cl^- E–2021, Automated Ferricyanide Method.* Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms a highly colored ferric thiocyanate, of which the intensity is proportional to the chloride concentration. The 2011 editorial revision currently is approved in Table IB for determination of chloride.

18. *4500 Series Cyanide Total or Available.*

a. *4500–CN^- B–2021, Manual Distillation (as Preliminary Treatment of Samples).* Total cyanides are measured after preliminary treatment of samples for preservation and to remove interferences. The preliminary treatment required depends on which interfering substances the samples contain. Distillation removes many interfering substances, but other pretreatment procedures will be needed for sample containing sulfides, fatty acids, oxidizing agents, nitrites, and nitrates. The 2016 version of the method currently is approved in Table IB for preliminary treatment of samples to be used for determination of cyanide.

b. *4500–CN^- C–2021, Total Cyanide after Distillation.* Hydrogen cyanide (HCN) is liberated from an acidified sample by distillation and purging with air, with the HCN gas collected in a NaOH scrubbing solution. The cyanide concentration in the scrubbing solution is determined via titrimetric, colorimetric, or potentiometric procedures. The 2016 version of the method currently is approved in Table IB for preliminary treatment of samples to be used for determination of cyanide.

c. *4500–CN^- D–2021, Titrimetric Method.* CN^- in the alkaline distillate from the preliminary treatment procedures (4500–CN^- B and C) is titrated with standard silver nitrate ($AgNO_3$) to form the soluble cyanide complex $Ag(CN)_2^-$. As soon as all CN^- has been complexed and a small excess of Ag^+ has been added, the silver-sensitive indicator, *p*-dimethylaminobenzalrhodanine, detects the excess Ag^+ and immediately changes color from yellow to salmon. The 2016 version of the method currently is approved in Table IB for determination of cyanide.

d. *4500–CN^- E–2021, Spectrophotometric Method.* Total CN^- in the alkaline distillate from the preliminary treatment procedures (4500–CN^- B and C) is converted to cyanogen chloride (CNCl) by reaction with chloramine-T at pH <8 without hydrolyzing to cyanate (CNO^-). After the reaction is complete, adding a pyridine-barbituric acid reagent turns CNCl a red-blue color. Maximum color absorbance in aqueous solution is between 575 and 582 nm. The 2016 version of the method currently is approved in Table IB for determination of cyanide.

e. *4500–CN^- F–2021, Ion Selective Electrode Method.* Total CN^- in the alkaline distillate from the preliminary treatment procedures (4500–CN^- B and C) is determined potentiometrically by using a CN^--ion selective electrode. The 2016 version of the method currently is approved in Table IB for determination of cyanide.

f. *4500–CN^- G–2021, Cyanides Amenable to Chlorination after Distillation.* Available cyanide, or cyanide amenable to chlorination (CATC), can be determined when 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. CATC is calculated by the difference between the results for cyanide in the unchlorinated sample and the results for the chlorinated sample. The 2016 version of the method currently is approved in Table IB for preliminary treatment of samples to be used for determination of available cyanide.

g. *4500–CN^- N–2021, Total Cyanide after Distillation by Flow Injection Analysis.* Total cyanides are digested and steam-distilled from the sample (4500–CN^- C), The cyanide in this distillate is converted to CNCl by reaction with chloramine-T at pH <8. The CNCl then forms a red-blue dye by reacting with pyridine-barbituric acid reagent. The absorbance of this red dye is measured at 570 nm and is proportional to the total or weak acid dissociable cyanide in the sample. The 2016 version of the method currently is approved in Table IB for determination of cyanide.

19. *4500 Total Fluoride Series.*

a. *4500–F^- B–2021, Preliminary Distillation Step.* Fluoride is separated from other nonvolatile constituents in water by conversion to hydrofluoric or fluosilicic acid and subsequent distillation. The conversion is accomplished by using a strong, high-boiling acid. To protect against glassware etching, hydrofluoric acid is converted to fluosilicic acid by using soft glass beads. Quantitative fluoride recovery is accomplished by using a relatively large sample. Acid and sulfate carryover are minimized by distilling over a controlled temperature range. The 2011 editorial revision currently is approved in Table IB for preliminary treatment of samples to be used for determination of fluoride.

b. *4500–F^- C–2021, Ion-Selective Electrode Method.* The fluoride electrode is an ion-selective sensor that measures the ion activity of fluoride in solution rather than concentration. The key element in the fluoride electrode is the laser-type doped lanthanum fluoride crystal across which a potential is established by fluoride solutions of different concentrations. The crystal contacts the sample solution at one face and an internal reference solution at the other. Fluoride ion activity depends on the solution total ionic strength and pH, and on fluoride complexing species. Adding an appropriate buffer provides a nearly uniform ionic strength background, adjusts pH, and breaks up complexes. In effect, the electrode measures concentration. The 2011 editorial revision currently is approved in Table IB for determination of fluoride.

c. *4500–F^- D–2021, SPADNS Method.* The SPADNS colorimetric method is based on the reaction between fluoride and a ''lake'' of zirconium-dye. Fluoride reacts with the dye lake, dissociating a portion of it into a colorless complex anion (ZrF_6^{2-}) and the dye. As the amount of fluoride increases, the color produced becomes progressively lighter and absorbance is measured colorimetrically at 570 nm. The 2011 editorial revision currently is approved in Table IB for determination of fluoride.

d. *4500–F^- E–2021, Complexone Method.* The sample is distilled in the automated system, and the distillate is reacted with alizarin fluorine blue-lanthanum reagent to form a blue complex that is measured colorimetrically at 620 nm. The 2011 editorial revision currently is approved in Table IB for determination of fluoride.

20. *4500 Hydrogen ion (pH). 4500–H^+ B–2021, Electrometric Method.* The basic principle of electrometric pH measurement is determination of the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. The hydrogen electrode consists of a platinum electrode across which hydrogen gas is bubbled at a pressure of 101 kilopascal. Because of difficulty in its use and the potential for poisoning the hydrogen electrode, the glass electrode commonly is used. The electromotive force (emf) produced in the glass electrode system varies linearly with pH. This linear relationship is described by plotting the measured emf against the pH of different buffers. A sample's pH is determined by extrapolation. This version of the method adds information to Section 2—Apparatus, regarding equipment that may be used for manual or automatic temperature compensation. The 2011 editorial revision currently is approved in Table IB for determination of pH.

21. *4500 Kjeldahl Nitrogen (TKN) Series.*

a. *4500–N_{org} B–2021, Macro-Kjeldahl Method.* In the presence of sulfuric acid (H_2SO_4), potassium sulfate (K_2SO_4), and a cupric sulfate ($CuSO_4$) catalyst, amino nitrogen of many organic materials is converted to ammonium. Free ammonia also is converted to ammonium. After the addition of base, the ammonia is distilled from an alkaline medium and absorbed in boric or sulfuric acid. The ammonia may be determined colorimetrically, by ammonia-selective electrode, or by titration with a standard mineral acid. The 2011 editorial revision currently is approved in Table IB for preliminary treatment of samples to be used for determination of total Kjeldahl nitrogen (TKN).

b. *4500–N_{org} C–2021, Semi-Micro-Kjeldahl Method.* This is a reduced-volume version of 4500 N_{org} B that specifies use of Kjeldahl flasks with a capacity of 100 mL in a semi-micro-Kjeldahl digestion apparatus equipped with heating elements to accommodate Kjeldahl flasks and a suction outlet to vent fumes. The 2011 editorial revision currently is approved in Table IB for preliminary treatment of samples to be used for determination of total Kjeldahl nitrogen (TKN).

c. *4500–N_{org} D–2021, Block Digestion and Flow Injection Analysis.* Samples are digested in a block digestor with sulfuric acid and copper sulfate as a catalyst. The digested sample is injected onto the FIA manifold, where its pH is controlled by raising it to a known, basic pH by neutralization with a concentrated buffer. This in-line neutralization converts the ammonium cation to ammonia, and also prevents undue influence of the sulfuric acid matrix on the pH-sensitive color reaction that follows. The ammonia thus produced is heated with salicylate and hypochlorite to produce a blue color that is proportional to the ammonia concentration. The color is intensified by adding sodium nitroprusside. The presence of EDTA in the buffer prevents the precipitation of calcium and magnesium. The resulting peak's absorbance is measured at 660 nm. The peak area is proportional to the concentration of total Kjeldahl nitrogen in the original sample. The 2011 editorial revision currently is approved in Table IB for determination of total Kjeldahl nitrogen.

22. *4500–NH_3 Nitrogen (Ammonia as nitrogen) Series*.

a. *4500–NH_3 B–2021, Preliminary Manual Distillation Step*. The sample is buffered at pH 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds. It is distilled into a solution of boric acid when titration is to be used, or into H_2SO_4, when the phenate method is used as the determinative step. The ammonia in the distillate can be determined either colorimetrically by the phenate method or titrimetrically with standard H_2SO_4 and a mixed indicator or a pH meter. Ammonia in the distillate also can be determined by the ammonia-selective electrode method, using 0.04 N H_2SO_4 to trap the ammonia. This revision replaces instructions for storage of ammonia-free water with instructions for preparation of ammonia-free water using an ion exchange resin and simply says that if high blank values are produced, the analyst should prepare fresh ammonia-free water. The 2011 editorial revision currently is approved in Table IB for preliminary treatment of samples to be used for determination of ammonia.

b. *4500–NH_3 C–2021, Titration Method*. The titrimetric method is used only on samples that have been carried through preliminary distillation. Ammonia is titrated with a standardized sulfuric acid titrant using a mixed indicator of methyl red and methylene blue. The 2011 editorial revision currently is approved in Table IB for determination of ammonia as well as for determination of total Kjeldahl nitrogen after appropriate digestion/distillation of the sample.

c. *4500–NH_3 D–2021, Electrode Method*. The ammonia-selective electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride. Dissolved ammonia ($NH_{3(aq)}$ and NH_4^+) is converted to $NH_{3(aq)}$ by raising the pH to above 11 with a strong base. $NH_{3(aq)}$ diffuses through the membrane and changes the internal solution pH that is sensed by a pH electrode. The fixed level of chloride in the internal solution is sensed by a chloride ion-selective electrode that serves as the reference electrode of the sample. Potentiometric measurements are made with a pH meter having an expanded millivolt scale or with a specific ion meter. The 2011 editorial revision currently is approved in Table IB for determination of ammonia, as well as for determination of total Kjeldahl nitrogen after appropriate digestion/distillation of the sample.

d. *4500–NH_3 E–2021, Electrode Method*. Ammonia is determined using an ammonia-selective electrode. When a linear relationship exists between concentration and response, known addition is convenient for measuring occasional samples because no calibration is needed. Because an accurate measurement requires that the concentration at least double as a result of the addition, sample concentration must be known within a factor of three. The total concentration of ammonia can be measured in the absence of complexing agents down to 0.8 mg/L NH_3-N or in the presence of a large excess (50 to 100 times) of complexing agent. The 2011 editorial revision currently is approved in Table IB for determination of ammonia, as well as for determination of total Kjeldahl nitrogen after appropriate digestion/distillation of the sample.

e. *4500–NH_3 F–2021, Phenate Method*. An intensely blue compound, indophenol, is formed by the reaction of ammonia, hypochlorite, and phenol catalyzed by sodium nitroprusside. The color is measured spectrophotometrically at 640 nm. The 2011 editorial revision currently is approved in Table IB for determination of ammonia, as well as for determination of total Kjeldahl nitrogen after appropriate digestion/distillation of the sample.

f. *4500–NH_3 G–2021, Semi-Automated Phenate Method*. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside. The color is measured spectrophotometrically at 630 to 660 nm. The 2011 editorial revision currently is approved in Table IB for determination of ammonia, as well as for determination of total Kjeldahl nitrogen after appropriate digestion/distillation of the sample.

g. *4500–NH_3 H–2021, Semi-Automated Phenate Method*. A water sample containing ammonia or ammonium cation is injected into an FIA carrier stream to which a complexing buffer (alkaline phenol) and hypochlorite are added. This reaction, the Berthelot reaction, produces the blue indophenol dye. The blue color is intensified by the addition of nitroferricyanide. The resulting peak's absorbance is measured at 630 nm. The peak area is proportional to the concentration of ammonia in the original sample. The 2011 editorial revision currently is approved in Table IB for determination of ammonia, as well as for determination of total Kjeldahl nitrogen after appropriate digestion/distillation of the sample.

23. *4500–NO_2^- Nitrite as Nitrogen.* 4500–NO_2^- B–2021, Spectrophotometric Method. Nitrite (NO_2^-) in a sample is determined through formation of a reddish-purple azo dye produced at pH 2.0 to 2.5 by coupling diazotized sulfanilamide with *N*-(1-naphthyl)-ethylenediamine dihydrochloride (NED) and absorbance is measured spectrophotometrically at 543 nm. The 2011 editorial revision currently is approved in Table IB for determination of nitrite.

24. *4500–NO_3^- Nitrogen (Nitrite/Nitrate as Nitrogen Series).*

a. *4500–NO_3^- D–2019, Nitrate Electrode Method*. Nitrate is measured using an ion-selective electrode that develops a potential across a thin, inert membrane holding in place a water-immiscible liquid ion exchanger. The 2016 version of the method currently is approved in Table IB for determination of nitrate.

b. *4500–NO_3^- E–2019, Cadmium Reduction Method*. Nitrate (NO_3^-) is reduced almost quantitatively to nitrite (NO_2^-) in the presence of cadmium (Cd). This method uses commercially available Cd granules treated with copper sulfate ($CuSO_4$) and packed in a glass column. The NO_2^- is then diazotized with sulfanilamide and coupled with NED to form a highly colored azo dye that is measured spectrophotometrically. To correct for any NO_2^- present in the sample before NO_3^- reduction, samples also must be analyzed without the reduction step. The 2016 version of the method currently is approved in Table IB for determination of nitrate (by subtraction), as well as for determination of combined nitrate + nitrite, and for determination of nitrite singly when bypassing the reduction step.

c. *4500–NO_3^- F–2019, Automated Cadmium Reduction Method*. This is an automated version of the cadmium reduction method 4500 NO_3^- E. Nitrate in a sample is reduced to nitrite using cadmium reduction and then diazotized with sulfanilamide and coupled with NED to form a highly colored azo dye that is measured spectrophotometrically. To correct for any NO_2^- present in the sample before NO_3^- reduction, samples also must be analyzed without the reduction step. The 2016 version of the method currently is approved in Table IB for determination of nitrate (by subtraction), as well as for determination of combined nitrate +

nitrite, and for determination of nitrite singly when bypassing the reduction step.

d. *4500–NO^{3-} H–2019, Automated Hydrazine Reduction Method.* Nitrate in a sample is reduced to nitrite using hydrazine sulfate then diazotized with sulfanilamide and coupled with NED to form a highly colored azo dye that is measured spectrophotometrically. The 2016 version of the method currently is approved in Table IB for determination of combined nitrate and nitrite.

e. *4500–NO^{3-} I–2019, Cadmium Reduction Flow Injection Method.* A sample is passed through a copperized cadmium column to quantitatively reduce its nitrate content to nitrite. The nitrite is diazotized with sulfanilamide and coupled with NED to yield a water-soluble dye with a magenta color whose absorbance at 540 nm is proportional to the nitrate + nitrite in the sample. Nitrite concentrations may be determined by bypassing the cadmium column and nitrate concentration may be calculated by subtraction of the result for the nitrite concentration from the result for the combined nitrate + nitrite concentration. The 2016 version of the method currently is approved in Table IB for determination of nitrate, as well as for determination of combined nitrate + nitrite, and for determination of nitrite singly by bypassing the reduction step.

25. *4500–O Oxygen (Dissolved) Series.*

a. *4500–O B–2021, Iodometric Methods.* A divalent manganese solution is added and then a strong alkali is added to a sample in a glass-stoppered bottle and dissolved oxygen (DO) rapidly oxidizes an equivalent amount of the dispersed divalent manganous hydroxide precipitate into higher-valency hydroxides. Oxidized manganese reverts to the divalent state in the presence of iodide ions in an acidic solution, liberating an amount of iodine equivalent to the original DO content. The iodine is then titrated with a standard thiosulfate solution. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

b. *4500–O C–2021, Azide Modification.* The sample is treated with manganous sulfate, potassium hydroxide, and potassium iodide (the latter two reagents combined in one solution) and finally sulfuric acid. The initial precipitate of manganous hydroxide, $Mn(OH)_2$, combines with the dissolved oxygen in the sample to form a brown precipitate, manganic hydroxide, $MnO(OH)_2$. Upon acidification, the manganic hydroxide forms manganic sulfate, which acts as an oxidizing agent to release free iodine from the potassium iodide. The iodine, which is stoichiometrically equivalent to the dissolved oxygen in the sample, is then titrated with sodium thiosulfate or phenylarsine oxide (PAO). The azide modification effectively removes nitrite interference, which is the most common interference in biologically treated effluents and incubated biochemical oxygen demand (BOD) samples. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

c. *4500–O D–2021, Permanganate Modification.* The permanganate modification is used only on samples containing Fe(II) (*e.g.,* acid mine water). Concentrated sulfuric acid, potassium permanganate in solution and potassium fluoride in solution are added to the sample. Enough $KMnO_4$ solution is added to obtain a violet tinge that persists for 5 minutes. 0.5 to 1.0 mL potassium oxalate solution is then added only until permanganate color is removed completely. From this point, the procedure closely parallels that in 4500–O C. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

d. *4500–O E–2021, Alum Flocculation Modification.* Samples high in suspended solids may consume appreciable quantities of iodine in acid solution. The interference due to solids may be removed by alum flocculation. Concentrated ammonium hydroxide and aluminum potassium sulfate solution are added to a sample. The sample is allowed to settle for about 10 min and the clear supernatant is siphoned into a 250- to 300-mL DO bottle until it overflows. From this point, the procedure closely parallels that in 4500–O C. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

e. *4500–O F–2021, Copper Sulfate-Sulfamic Acid Flocculation Modification.* This modification is used for biological flocs (*e.g.,* activated sludge mixtures), which have high oxygen utilization rates. A copper sulfate-sulfamic acid inhibitor solution is added to the sample. The suspended solids are allowed to settle, and the relatively clear supernatant liquor is siphoned into a 250- to 300-mL DO bottle. From this point, the procedure closely parallels that in 4500–O C. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

f. *4500–O G–2021, Electrode Method.* Oxygen-sensitive polarographic or galvanic membrane electrodes are composed of two solid metal electrodes in contact with supporting electrolyte separated from the test solution by a selective membrane. Polyethylene and fluorocarbon membranes are commonly used because they are permeable to molecular oxygen and are relatively rugged. The diffusion current is linearly proportional to the molecular-oxygen concentration. The measured current can be converted easily to concentration units (*e.g.,* mg/L) by a number of calibration procedures. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

g. *4500–O H–2021, Luminescence-based Method.* The optical probe uses luminescence-based oxygen sensors to measure the light-emission characteristics of a luminescent reaction; oxygen quantitatively quenches the luminescence. The change in the luminescence signal's lifetime correlates to the DO concentration. The 2016 version of the method currently is approved in Table IB for determination of dissolved oxygen.

26. *4500–P Phosphorus Total and Ortho Phosphorus Series.*

a. *4500–P B–2021, Digestion Sample Preparation.* Because phosphorus may occur in combination with organic matter, a digestion method to determine total phosphorus must be able to oxidize organic matter effectively to release phosphorus as orthophosphate. Three digestion methods are given in 4500–P B.3, 4, and 5. The perchloric acid method in B.5 is the most vigorous and time-consuming method, and is recommended for particularly difficult samples, such as sediments. The nitric acid-sulfuric acid method is recommended for most samples. The simplest digestion method that may be used for determination of total phosphorus is the persulfate oxidation technique in which 50 mL of an unfiltered sample is boiled with sulfuric acid and either ammonium persulfate or potassium persulfate for approximately 30–40 minutes or until a final volume of about 10 mL is reached. The 2011 editorial revision is currently approved in Table IB for preliminary treatment of samples to be used for determination of total phosphorus as orthophosphorus using manual or automated versions of the ascorbic acid reduction, colorimetric methods.

b. *4500–P E–2021, Manual Method.* Ammonium molybdate and antimony potassium tartrate react in an acid medium with orthophosphate to form phosphomolybdic acid, a heteropoly acid that is reduced to intensely colored molybdenum blue by ascorbic acid and is measured spectrophotometrically. This revision adds that possible interference from silicate should be evaluated when reporting concentrations less than 10 µg/L. The 2011 editorial revision currently is approved in Table IB for determination of total phosphorus after digestion of the sample, as well as for determination of orthophosphorus in a filtered, undigested sample.

c. *4500–P F–2021, Automated Ascorbic Acid Reduction Method.* Ammonium molybdate and antimony potassium tartrate react with orthophosphate in an acid medium to form an antimony-phosphomolybdate complex, which on reduction with ascorbic acid yields an intense blue color suitable for photometric measurement using continuous flow analytical equipment. The 2011 editorial revision currently is approved in Table IB for determination of total phosphorus after digestion of the sample, as well as for determination of orthophosphorus in a filtered, undigested sample.

d. *4500–P G–2021, Automated.* Ammonium molybdate and antimony potassium tartrate react with orthophosphate in an acid medium to form an antimony-phosphomolybdate complex, which on reduction with ascorbic acid yields an intense blue color suitable for photometric measurement using flow injection analysis. The 2011 editorial revision currently is approved in Table IB for determination of total phosphorus after digestion of the sample as well, as for determination of orthophosphorus in a filtered, undigested sample.

e. *4500–P H–2021, Automated Total Phosphorus.* Samples are manually digested using the approved procedure for preliminary treatment of samples to be used for determination of total phosphorus. When the resulting solution is injected onto the manifold, the orthophosphate ion reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex. This complex is reduced with ascorbic acid to form a blue complex suitable for photometric measurement using flow injection analysis. The 2011 editorial revision currently is approved in Table IB for determination of total phosphorus.

27. *4500–S^{2-} Sulfide Series.*

a. *4500–S^{2-} B–2021, Sample Pretreatment.* Dissolved sulfide is measured by first removing insoluble matter. This is done by adding sodium hydroxide and aluminum chloride solutions producing an aluminum hydroxide floc that is settled, leaving a clear supernatant for analysis. The 2011 editorial revision currently is approved in Table IB for preliminary treatment of samples to be used for determination of sulfide.

b. *4500–S^{2-} C–2021, Sample Pretreatment.* Interferences due to sulfite, thiosulfate, iodide, and many other soluble substances, but not ferrocyanide, are eliminated by first precipitating zinc sulfide (ZnS) by addition of sodium hydroxide and zinc acetate solutions, removing the supernatant, and replacing it with reagent water. The same procedure is used even when not needed for removal of interferences, to concentrate sulfide prior to analysis. The 2011 editorial revision currently is approved in Table IB for preliminary treatment of samples to be used for determination of sulfide.

c. *4500–S^{2-} D–2021, Colorimetric Method.* The methylene blue method is based on the reaction of sulfide, ferric chloride, and dimethyl-*p*-phenylenediamine to produce methylene blue. Ammonium phosphate is added after color development to remove ferric chloride color, which is measured photometrically. The procedure is applicable at sulfide concentrations between 0.1 and 20.0 mg/L. There are no other procedural changes. The 2011 editorial revision currently is approved in Table IB for determination of sulfide.

d. *4500–S^{2-} F–2021, Titrimetric.* Iodine oxidizes sulfide in acid solution. A titration based on this reaction is an accurate method for determining sulfide at concentrations above 1 mg/L if interferences are absent and if loss of H_2S is avoided. The 2011 editorial revision currently is approved in Table IB for determination of sulfide.

e. *4500–S^{2-} G–2021, Ion-Selective Electrode Method.* The potential of a sulfide ion-selective electrode (ISE) is related to the sulfide ion activity. An alkaline antioxidant reagent (AAR) is added to samples and standards to inhibit oxidation of sulfide by oxygen and to provide a constant ionic strength and pH. Use of the AAR allows calibration in terms of total dissolved sulfide concentration. All samples and standards must be at the same temperature. Sulfide concentrations between 0.032 mg/L and 100 mg/L can be measured without preconcentration. For lower concentrations, preconcentration is necessary. The 2011 editorial revision currently is approved in Table IB for determination of sulfide.

28. *4500–SiO_2 Silica Series.*

a. *4500–SiO_2 C–2021, Colorimetric Method.* Ammonium molybdate at pH approximately 1.2 reacts with silica and any phosphate present to produce heteropoly acids. Oxalic acid is added to destroy the molybdophosphoric acid, but not the molybdosilicic acid. Even if phosphate is known to be absent, the addition of oxalic acid is highly desirable and is a mandatory step. The intensity of the yellow color produced is proportional to the concentration of molybdate-reactive silica and is measured photometrically. The 2011 editorial revision currently is approved in Table IB for determination of silica.

b. *4500–SiO_2 E–2021, Automated Method for Molybdate-Reactive Silica.* Ammonium molybdate at pH approximately 1.2 reacts with silica and any phosphate present to produce heteropoly acids. Oxalic acid is added to destroy the molybdophosphoric acid, but not the molybdosilicic acid. The yellow molybdosilicic acid is reduced by means of amino naphthol sulfonic acid to heteropoly blue. The blue color is more intense than the yellow color of 4500–SiO_2 C and provides increased sensitivity. The 2011 editorial revision currently is approved in Table IB for determination of silica.

c. *4500–SiO_2 F–2021, Automated Method for Molybdate-Reactive Silicate.* Silicate reacts with molybdate under acidic conditions to form yellow beta-molybdosilicic acid. This acid is subsequently reduced with stannous chloride to form a heteropoly blue complex that is measured photometrically. Oxalic acid is added to reduce the interference from phosphate. The 2011 editorial revision currently is approved in Table IB for determination of silica.

29. *4500–SO_4^{2-} Sulfate Series.*

a. *4500–SO_4^{2-} C–2021, Gravimetric Method with Ignition of Residue.* Sulfate is precipitated in a hydrochloric acid (HCl) solution as barium sulfate ($BaSO_4$) by the addition of barium chloride ($BaCl_2$). The precipitation is carried out near the boiling temperature, and after a period of digestion, the precipitate is filtered, washed with water until free of Cl^-, ignited at 800 °C for an hour and weighed as $BaSO_4$. The 2011 editorial revision currently is approved in Table IB for determination of sulfate.

b. *4500–SO_4^{2-} D–2021, Gravimetric Method with Drying of Residue.* Sulfate is precipitated in a hydrochloric acid (HCl) solution as barium sulfate ($BaSO_4$) by the addition of barium chloride ($BaCl_2$). The precipitation is carried out near the boiling temperature, and after a period of digestion the precipitate is filtered, washed with water until free of Cl^-, dried to a constant weight in an oven at 105 °C or higher, and weighed as $BaSO_4$. The 2011 editorial revision currently is approved in Table IB for determination of sulfate.

c. *4500–SO_4^{2-} E–2021, Turbidimetric Method.* Sulfate ion (SO_4^{2-}) is precipitated in an acetic acid medium with barium chloride ($BaCl_2$) to form barium sulfate ($BaSO_4$) crystals of uniform size. Light absorbance of the $BaSO_4$ suspension is measured by a photometer and the SO_4^{2-} concentration is determined by comparison of the reading with a standard curve. The 2011 editorial revision currently is approved in Table IB for determination of sulfate.

d. *4500–SO_4^{2-} F–2021, Automated Colorimetric Method.* Barium sulfate is formed by the reaction of the SO_4^{2-} with barium chloride ($BaCl_2$) at a low pH. At high pH, excess barium reacts with methylthymol blue (MTB) to produce a blue chelate. The uncomplexed methylthymol blue is gray. The intensity of gray (uncomplexed methylthymol blue) is measured photometrically and is proportional to concentration of sulfate. The 2011 editorial revision currently is approved in Table IB for determination of sulfate.

e. *4500–SO_4^{2-} G–2021, Automated Colorimetric Method.* At pH 13.0, barium forms a blue complex with MTB. The sample is injected into a low, but known, concentration of sulfate. The sulfate from the sample then reacts with the ethanolic barium-MTB solution and displaces the MTB from the barium to give barium sulfate and uncomplexed MTB. Uncomplexed MTB has a grayish color. The pH is raised with NaOH and the gray color of the uncomplexed MTB is measured photometrically. The intensity of the gray color is proportional to the sulfate concentration. The 2011 editorial revision currently is approved in Table IB for determination of sulfate.

30. *Sulfite 4500–SO_3^{2-} B–2021, Titrimetric Iodometric Method.* An acidified sample containing sulfite (SO_3^{2-}) is titrated with a standardized potassium iodide-iodate titrant. Free iodine, liberated by the iodide-iodate reagent, reacts with SO_3^{2-}. The titration endpoint is signaled by the blue color resulting from the first excess of iodine reacting with a starch indicator. The 2011 editorial revision currently is approved in Table IB for determination of sulfite.

31. *5520 Oil and Grease Series.*

a. *5520 B–2021, Liquid-Liquid, Partition-Gravimetric Method.* Dissolved or emulsified oil and grease is extracted from water by intimate contact with an extracting solvent (n-hexane). The extract is dried over sodium sulfate. The solvent is then distilled from the extract and the hexane extractable material is desiccated and weighed. Some extractables, especially unsaturated fats and fatty acids, oxidize readily; hence, special precautions regarding temperature and solvent vapor displacement are included to minimize this effect. Organic solvents shaken with some samples may form an emulsion that is very difficult to break. This method includes a means for handling such emulsions. Recovery of solvents is discussed. Solvent recovery can reduce both vapor emissions to the atmosphere and costs. The 2011 editorial revision currently is approved in Table IB for determination of oil and grease (hexane extractable material or HEM).

b. *5520 F–2021, Hydrocarbons.* The oil and grease extracted by 5520 B is used for this test. When only hydrocarbons are of interest, this procedure is introduced before final measurement. When hydrocarbons are to be determined after total oil and grease has been measured, redissolve the extracted oil and grease in n-hexane. Silica gel has the ability to adsorb polar materials. The solution of extracted hydrocarbons and fatty materials in n-hexane is mixed with silica gel, and the fatty acids are removed selectively from solution. The solution is filtered to remove the silica gel, the solvent is distilled, and the silica gel treated hexane extractable material (SGT–HEM) is weighed. The materials not eliminated by silica gel adsorption are designated hydrocarbons by this test. The 2011 editorial revision currently is approved in Table IB for determination of oil and grease (hexane extractable material or HEM).

32. *5530 Phenols Series.*

a. *5530 B–2021, Manual Distillation.* Phenols, defined as hydroxy derivatives of benzene and its condensed nuclei, may occur in domestic and industrial wastewaters, natural waters, and potable water supplies. Phenols are distilled from nonvolatile impurities. Because the volatilization of phenols is gradual, the distillate volume must ultimately equal that of the original sample. The 2010 version of the method currently is approved in Table IB for preliminary treatment of samples to be used for determination of phenols.

b. *5530 D–2021, Colorimetric Method.* Steam-distillable phenolic compounds react with 4-aminoantipyrine at pH 7.9 ± 0.1 in the presence of potassium ferricyanide to form a colored antipyrine dye. This dye is kept in aqueous solution and the absorbance is measured photometrically at 500 nm. The 2010 version of the method currently is approved in Table IB for determination of phenol. Note that for regulatory compliance monitoring required under the Clean Water Act, the colorimetric reaction must be performed at a pH of 10.0 ± 0.2 as stated in 40 CFR 136.3, Table IB, footnote 27.

33. *5540 Surfactants.*

5540 C–2021. This colorimetric method comprises three successive extractions from an acid aqueous medium containing excess methylene blue into chloroform ($CHCl_3$), followed by an aqueous backwash and measurement of the blue color in the $CHCl_3$ by spectrophotometry at 652 nm. The method is applicable at methylene blue active substances concentrations down to about 0.025 mg/L. The 2011 editorial revision currently is approved in Table IB for determination of surfactants.

34. *6200 Volatile Organic Compounds Series.*

a. In the 6200 B–2020, Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric (GC/MS) Method, volatile organic compounds are transferred efficiently from the aqueous to the gaseous phase by bubbling an inert gas (*e.g.,* helium) through a water sample contained in a specially designed purging chamber at ambient temperature. The vapor is swept through a sorbent trap that adsorbs the analytes of interest. After purging is complete, the trap is heated and back-flushed with the same inert gas to desorb the compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the compounds. The detector is a mass spectrometer. The 2011 editorial revision currently is approved in Table IC for determination of benzene, bromodichloromethane, bromoform, bromomethane, carbon tetrachloride, chlorobenzene, chloroethane, chloroform, chloromethane, dibromochloromethane, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, dichlorodifluoromethane, 1,1-dichloroethane, 1,2-dichloroethane, 1,1-dichloroethene, *trans*-1,2-dichloroethene, 1,2-dichloropropane, *cis*-1,3-dichloropropene, *trans*-1,3-dichloropropene, ethylbenzene, methylene chloride, 1,1,2,2-tetrachloroethane, tetrachloroethene, toluene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethene, trichlorofluoromethane, and vinyl chloride.

b. *6200 C–2020, Purge and Trap Capillary-Column Gas Chromatographic (GC) Method.* Volatile organic compounds are transferred efficiently from the aqueous to the gaseous phase by bubbling an inert gas (*e.g.,* helium) through a water sample contained in a specially designed purging chamber at ambient temperature. The vapor is swept through a sorbent trap that adsorbs the analytes of interest. After purging is complete, the trap is heated and back-flushed with the same inert gas to desorb the compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the compounds and detected using a photoionization detection and an electrolytic conductivity detection in series. The 2011 editorial revision currently is approved in Table IC for determination of benzene, bromodichloromethane, bromoform, bromomethane, carbon tetrachloride, chlorobenzene, chloroethane, chloroform, chloromethane, dibromochloromethane, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,1-dichloroethane, 1,2-dichloroethane, 1,1-dichloroethene, *trans*-1,2-dichloroethene, 1,2-dichloropropane, *cis*-1,3-dichloropropene, *trans*-1,3-dichloropropene, ethylbenzene, methylene chloride, 1,1,2,2-tetrachloroethane, tetrachloroethene, toluene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethene, trichlorofluoromethane, and vinyl chloride.

35. *6410 Extractable Base/Neutrals and Acids.*

6410 B–2020, Liquid-Liquid Extraction Gas Chromatographic/Mass Spectrometric Method. This method is applicable to the determination of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography in municipal and industrial discharges. A measured volume of sample is extracted serially with methylene chloride at a pH of approximately 2 and again at pH 11. The extract is dried, concentrated, and analyzed by GC/MS. Qualitative compound identification is based on retention time and relative abundance of three characteristic masses (*m/z*). Quantitative analysis uses internal-standard techniques with a single characteristic *m/z.* This revision adds a note that although the method was validated extracting base neutrals first and then acids, performance may be improved by extracting acids first and then base neutrals. In addition, EPA proposes to approve method 6410–B for endrin aldehyde in Table ID. This parameter was inadvertently left off the 2000 MUR rulemaking. The 2000 version of the method currently is approved in Table IC for determination of acenaphthene, acenaphthylene, anthracene, benzidine, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, butyl benzyl phthalate, bis(2-chloroethoxy) methane, bis(2-chloroethyl) ether, bis(2-ethylhexyl) phthalate, bromodichloromethane, 4-bromophenyl phenyl ether, 4-chloro-3-methyl phenol, 2-chloronaphthalene, 2-chlorophenol, 4-chlorophenyl phenyl ether, chrysene, dibenzo(a,h)anthracene, 3,3′-dichlorobenzidine, 2,4-dichlorophenol, diethyl phthalate, 2,4-dimethylphenol, dimethyl phthalate, di-*n*-butyl phthalate, di-*n*-octyl phthalate, 2,4-dinitrophenol, 2,4-dinitrotoluene, 2,6-dinitrotoluene, fluoranthene, fluorene, hexachlorobenzene, hexachlorobutadiene, hexachlorocyclopentadiene, indeno(1,2,3-c,d) pyrene, isophorone, 2-methyl-4,6-dinitrophenol, naphthalene, nitrobenzene, 2-nitrophenol, 4-nitrophenol, n-nitrosodi-*n*-propylamine, n-nitrosodiphenylamine, PCB–1016, PCB–1221, PCB–1232, PCB–1242, PCB–1248, PCB–1254, PCB–1260, pentachlorophenol, phenanthrene, phenol, pyrene, 1,2,4-trichlorobenzene, and 2,4,6-trichlorophenol and in Table ID for determination of aldrin, α-BHC, β-BHC, δ-BHC, γ-BHC (lindane), chlordane, 4,4′-DDD, 4,4′-DDE, 4,4′-DDT, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, heptachlor, heptachlor epoxide, and toxaphene.

36. *6420 Phenols.*

6420 B–2020, Liquid-Liquid Extraction Gas Chromatographic Method. A measured volume of sample is acidified and extracted with methylene chloride. The extract is dried and exchanged to 2-propanol during concentration. Target analytes in the extract are separated by gas chromatography and are identified by retention time and measured with a flame ionization detector, or derivatized and measured with an electron capture detector. This revision of the method replaces distilled, deionized water with reagent water, adds that the packed columns used for validation of the method are no longer available or recommended, and includes information on alternative capillary columns that may be used. The 2000 version of the method currently is approved in Table IC for determination of 4-chloro-3-methylphenol, 2-chlorophenol, 2,4-dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol, 2-nitrophenol, 4-nitrophenol, pentachlorophenol, phenol, and 2,4,6-trichlorophenol.

37. *6440 Polynuclear Aromatic Hydrocarbons.*

6440 B–2021, Liquid-Liquid Extraction Chromatographic Method. A measured volume of sample is extracted with methylene chloride. The extract is dried, concentrated, and separated by the high-performance liquid chromatographic (HPLC) or gas chromatographic (GC) method. Ultraviolet (UV) and fluorescence detectors are used with HPLC to identify and measure the polynuclear aromatic hydrocarbons. A flame ionization detector is used with GC. The 2005 version of the method currently is approved in Table IC for determination of acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-c,d)pyrene, naphthalene, phenanthrene, and pyrene.

38. *6630 Organochlorine Pesticides Series.*

a. 6630 B–2021, Liquid-Liquid Extraction Gas Chromatographic Method I, in this procedure, the pesticides are extracted with a mixed solvent, diethyl ether-hexane or methylene chloride-hexane, by either liquid-liquid extraction using a separatory funnel or by continuous liquid-liquid extraction. The extract is concentrated by evaporation and, if necessary, is cleaned up by column adsorption chromatography. The individual pesticides then are separated by gas chromatography and the compounds are measured with an electron capture detector (ECD). This revision of the method adds information regarding alternative capillary columns that may be used in place of the packed columns that were used for validation of the method, removes information regarding preparation of packed columns, replaces information regarding manual injection technique with use of an autosampler and states that gas chromatography/mass spectrometry (GC/MS) may be used for confirmatory analyses in place of a second column and ECD detection. There are no other procedural changes. The 2007 version of the method currently is approved in Table ID for determination of aldrin, α-BHC, β-BHC, δ-BHC, γ-BHC (lindane), captan, carbophenothion, chlordane, 4,4′-DDD, 4,4′-DDE, 4,4′-DDT, dichloran, dieldrin, endosulfan I, endosulfan II, endrin, heptachlor, heptachlor epoxide, isodrin, malathion, methoxychlor, mirex, parathion methyl, parathion ethyl, PCNB, strobane, toxaphene, and trifluralin.

b. In 6630 C–2021, Liquid-Liquid Extraction Gas Chromatographic Method II, a measured volume of sample is extracted with methylene chloride either by liquid-liquid extraction using separatory funnels or by continuous liquid-liquid extraction. The extract is dried and exchanged to hexane during concentration. The target analytes are separated by gas chromatography and the compounds are measured with an electron capture detector (ECD). This revision of the method adds information regarding alternative capillary columns that may be used in place of the packed columns that were used for validation of the method, and states that gas chromatography/mass spectrometry (GC/MS) may be used for confirmatory analyses in place of a second column and ECD detection. There are no other procedural changes. The 2007 version of the method currently is approved in Table ID for determination of aldrin, α-BHC, β-BHC, δ-BHC, γ-BHC (lindane), chlordane, 4,4′-DDD, 4,4′-DDE, 4,4′-DDT, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide, isodrin, methoxychlor, mirex, PCNB, strobane, and toxaphene.

39. *6640 Acidic Herbicide Compounds.*

6640 B–2021, Micro Liquid-Liquid Extraction Gas Chromatographic Method. A 40-mL sample is adjusted to pH ≥12 with 4 N sodium hydroxide and is kept for 1 hour at room temperature to hydrolyze derivatives. Because the chlorphenoxy acid herbicides are formulated as a variety of esters and salts, the hydrolysis step is required and may not be skipped. The aqueous sample then is acidified with sulfuric acid to pH ≤1 and extracted with 4 mL of methyl *tert*-butyl ether (MtBE) that contains the internal standard. The chlorinated acids, which have been partitioned into the MtBE, then are converted to methyl esters by derivatization with diazomethane. The target esters are separated and detected by capillary column gas chromatography using an electron capture detector (GC/ECD). Analytes are quantified using an internal-standard-based calibration curve. The 2006 editorial revision currently is approved in Table IC for determination of 2,4–D, 2,4,5–T, and 2,4,5–TP (Silvex).

D. Changes to 40 CFR 136.3 To Include Alternate Test Procedures in Table IC

To promote method innovation, 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 alternate test procedure (ATP) program is described for CWA applications at 40 CFR 136.4 and 136.5. EPA is proposing two ATPs for nationwide use. Based on EPA's review, the performance of these ATPs is equally effective as other methods already approved for measurement of 2,3,7,8-substituted tetra- through octa-chlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDDs/PCDFs) in wastewater. The ATP applicants supplied EPA with study reports that contain the data from their validation studies. These study reports, the final methods, and the letters documenting EPA's review are included as supporting documents in the docket for this proposed rule.

These proposed new methods are: SGS AXYS Method ATM 16130, ''Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs) Using Waters and Agilent Gas Chromatography-Tandem-Mass Spectrometry (GC/MS/MS), Revision 1.0 and Pace Analytical Method PAM–16130–SSI, ''Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs) Using Shimadzu Gas Chromatography Mass Spectrometry (GC–MS/MS), Revision 1.1.'' These ATPs are the results of separate collaborative efforts between SGS AXYS Analytical Services Ltd, and the instrument manufacturers Waters Corporation, Agilent Technologies, and between Pace Analytical Services LLC and the instrument manufacturer Shimadzu Scientific Instruments, Inc. These final methods are heavily adapted from Method 1613B. Neither ATP makes changes to the extraction or cleanup procedures specified in Method 1613B. All required quality control tests (or analogous tests) and associated QC acceptance criteria have been included in both SGS AXYS 16130 and PAM–16130–SSI.

To minimize costs to both the applicants and the Agency where possible, SGS AXYS, Pace Analytical, and the instrument manufacturers who collaborated on these methods worked closely with EPA's CWA ATP Coordinator to design single-laboratory validation studies for these methods. The goal of these validation studies was to demonstrate that all of the performance criteria specified in Method 1613B could be met and that comparable performance could be achieved when using GC–MS/MS instrumentation for determination of PCDDs/PCDFs in extracts from real-world samples.

EPA Method 1613B was promulgated at 40 CFR 136 in 1995 and remains the only approved method for dioxins and furans at NPDES permit levels (Methods 613 and 625.1 may only be used for screening). Method 1613B is also the only method approved at 40 CFR part 136 that relies on gas chromatography-high resolution mass spectrometry (GC/HRMS) as the determinative technique. As a result, the need for GC/HRMS instruments is somewhat limited, and market forces have led some instrument vendors to move away from supporting new GC/HRMS instrumentation. In addition, in the last 30 years, there has been substantial consolidation of manufacturers, with the disappearance of many of the vendors whose instruments were used to develop and validate Method 1613B.

In these two methods, referred to in the rule as ATM 16130 and PAM 16130–SSI, each sample is spiked with the same suite of carbon-13 labeled standards prior to extraction and those standards are used for isotope dilution quantitation in the same way as is done in EPA Method 1613B. All of the relevant QC acceptance criteria are the same in the methods as well. The difference between these methods and the approved EPA method is the use of an MS/MS detector system that uses Multiple Reaction Monitoring (MRM) in place of a high resolution mass spectrometer (HRMS) detector system. The GC portions of the methods did not change.

E. Corrections or Amendments to the Text and Tables of 40 CFR Part 136

In addition to the method revisions discussed in Section II.C of this preamble, Standard Methods has revised certain of their general quality control sections (2020, 3020, 4020 and 5020). EPA is proposing to update the year of the current references to these sections in 136.3 Table IB footnote 85, as well as add a reference to an additional Standard Methods Quality Control Section: Part 6000 Individual Organic Compounds, 6020, based on EPA's review of these sections. These Quality Control Standards are available for download at *www.standardmethods.org* at no charge. Further, during the preparation of this proposed rulemaking, EPA identified several minor errors or inconsistencies in the tables of approved methods. Therefore, EPA is proposing the following changes to 40 CFR 136.3, Tables IA, IB, IC or ID:

1. *Table IA.* Removing the units of ''number per 100 mL'' under parameter 1. Coliform (fecal), because parameter 1 is specifically for biosolids that are reported as ''number per gram dry weight''.

2. *Table IA.* Moving USGS Method ''B–0050–85'' from parameter 1. Coliform (fecal) number per gram dry weight to parameter 2. Coliform (fecal) number per 100 mL, to address an error from the previous rulemaking when Parameter 1 Coliform (fecal) was split

into two parameters to eliminate confusion as to which methods were approved for biosolids.

3. *Table IA.* Moving the phrase ''two-step'' in parameter 3, in the ''Method'' column from the second to the third line which returns the phrase to the proper line after having been inadvertently moved.

4. *Table IB.* Revising footnote 85 to remove bullet formatting.

5. *Table IB.* EPA proposes adding footnote 86 to Method 419D, listed as an approved method for determination nitrate using Colorimetric (Brucine sulfate) methodology. This addition corrects a long-standing typographical error regarding the appropriate footnote for this method in Table IB.

6. *Table IB.* Correcting an inadvertent error to footnote 57. The reference number was incorrectly changed to 335.4–1. The correct number is 335.4.

7. *Tables IC and ID.* Proposes adding footnote 15 to the Standard Method Column header and adding footnote 15 to refer to Quality Control Section: Part 6000 Individual Organic Compounds, 6020 (2019).

8. *Table IC.* The parameter 39, dichlorodifluoromethane, should refer to Method 6200 B rather than 6200 C for the GC/MS method.

9. *Table IC.* Parameters 66–72, 95, 96 and 97. These parameters are missing the footnote 10 that was inadvertently dropped in an earlier rulemaking. Footnote 10 to table IC applies to all of the 17 dioxin and furan congeners.

10. *Table IH.* Parameter 2 has method B–0025–85 is moved down one row because it was inadvertently moved. This method is a one-step membrane filtration (MF) method rather than a most probable number (MPN) method.

11. Footnote 5 to Table II for the preservation and holding time requirements for cyanide to add the year (2015) of the ASTM method D7365–09a (15). This practice is applicable for the collection and preservation of water samples for the analysis of cyanide. Samples are collected in appropriate containers and mitigated for known interferences either in the field during sample collection or in the laboratory prior to analysis. The sampling, preservation and mitigation of interference procedures described in this practice are recommended for the analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by ASTM Methods D2036, D4282, D4374, D6888, D6994, D7237, D7284, and D7511.

The recommended sampling and preservation procedures in the ASTM method have not changed since 2009, but the change to footnote 5 will simplify identification of the current method that is available from ASTM International. The 2015 reapproval date was already updated in footnote 6 to Table II in the 2021 methods update rule; however, adding the reapproval date was overlooked in the IBR section and in footnote 5 to Table II.

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

EPA is proposing adding five new methods in furtherance of the National Technology Transfer and Advancement Act of 1995 (NTTAA), Public Law 104–113, that provides 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 were submitted by Standard Methods and are consistent with other already approved methods. EPA is adding 4500–CN^{-} P–2021, 4500–CN^{-} Q–2021, 4500 CN^{-} R–2021, 4500–F^{-} G–2021 to Table IB for cyanide and fluoride and is adding 5520 G–2021 to Table IB for oil and grease, based on the following reasons:

1. *Cyanide.* Although method 4500–CN^{-} P–2021, Total Cyanide by Segmented Flow Injection, UV-Irradiation with Gas Diffusion, and Amperometric Measurement is new to *Standard Methods for the Examination of Water and Wastewater,* it is based on ASTM D7511–12(17), which is approved in Table IB for determination of total cyanide and relies on the same underlying chemistry and determinative technique to determine total cyanide. Total cyanide consists of dissolved HCN, sodium cyanide (NaCN), and various metal-cyanide complexes, which a continuous flow analyzer converts to aqueous HCN by mixing it with sulfuric acid, irradiating with UV light, and precipitating potentially interfering sulfides with bismuth ion. The aqueous HCN is captured in a donor stream that is passed across a hydrophobic gas-permeable membrane, which selectively diffuses the gaseous HCN into a parallel acceptor stream of dilute sodium hydroxide forming dissolved CN^{-}. The cyanide ion in this acceptor stream is measured using an amperometric detector, where the cyanide ion dissolves the silver electrode, resulting in a proportional current.

2. *4500–CN^{-} Q–2021, Weak and Dissociable Cyanide by Flow Injection, Gas Diffusion, and Amperometric Measurement.* Weak and dissociable cyanide consists of dissolved HCN, NaCN, and various metal-cyanide complexes and includes the same forms of cyanide as those measured using other methods approved in Table IB for determination of available cyanide. Analysts pretreat for weak and dissociable cyanide by mixing a sample with ligand reagents. They then inject the sample into a sulfuric acid and bismuth nitrate solution to produce a donor stream containing aqueous dissolved HCN and precipitated sulfide, if sulfide is present. The donor stream is passed across a hydrophobic gas-permeable membrane, which selectively diffuses gaseous HCN into a parallel acceptor stream of dilute sodium hydroxide, forming dissolved CN^{-}. The cyanide ion in this acceptor stream is measured using an amperometric detector, where the cyanide ion dissolves the silver electrode, resulting in a proportional current. Although this method is new to *Standard Methods for the Examination of Water and Wastewater,* it is based on ASTM D6888–16, which is approved in Table IB for determination of available cyanide and relies on the same underlying chemistry and determinative technique to determine available cyanide.

3. *4500–CN^{-} R–2021, Free Cyanide by Flow Injection, Gas Diffusion, and Amperometric Measurement.* Free cyanide (FCN) consists of dissolved HCN, NaCN, and the soluble fraction of various metal-cyanide complexes. To determine FCN, analysts pretreat a sample by mixing it with a buffered solution in the pH range of 6 to 8 that simulates the receiving water resulting in a donor stream containing aqueous dissolved HCN in equilibrium with the cyanide anion. The donor stream is passed across a hydrophobic gas-permeable membrane, which selectively diffuses gaseous HCN into a parallel acceptor stream that consists of dilute sodium hydroxide, forming dissolved CN^{-}. The cyanide ions in this acceptor stream are measured when it is passed through an amperometric detector, where the cyanide ion dissolves the silver electrode, resulting in a proportional current. Although this method is new to *Standard Methods for the Examination of Water and Wastewater,* it is based on ASTM D7237–15, which is approved in Table IB for determination of free cyanide and relies on the same underlying chemistry and determinative technique to determine free cyanide.

4. *Fluoride.* 4500–F^{-} G–2021, Ion-Selective Electrode Flow Injection Analysis is an automated version of method 4500–F^{-} C and relies on the same underlying chemistry and

determinative technique as USGS Method I–4237–85, which currently is approved in Table IB for determination of fluoride. Fluoride is determined potentiometrically by using a combination fluoride ion selective electrode (ISE) in a flow cell. The fluoride electrode consists of a lanthanum fluoride crystal across which a potential is developed by fluoride ions.

5. *Oil and Grease.* In 5520 G–2021, Solid-Phase, Partition-Gravimetric Method, dissolved or emulsified oil and grease is extracted from water by passing a sample through a solid-phase extraction (SPE) disk where the oil and grease are adsorbed by the disk and subsequently eluted with n-hexane. SPE is a modification allowed under EPA Methods 1664 A and B and relies on the same underlying chemistry and determinative technique as Methods 1664 A and B. Some extractables, especially unsaturated fats and fatty acids, oxidize readily; hence, special precautions regarding temperature and solvent vapor displacement are provided. This method is not applicable to materials that volatilize at temperatures below 85 °C, or crude and heavy fuel oils containing a significant percentage of material not soluble in n-hexane. This method may be a satisfactory alternative to liquid-liquid extraction techniques, especially for samples that tend to form difficult emulsions during the extraction step.

IV. Incorporation 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 parameter (or 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 (see Sections III.B, III.C, and III.F of this preamble). 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 of this preamble). The proposed copyrighted VCSB methods are available on their respective websites (*standardmethods.org* and *astm.org*) to everyone at a cost determined by the VCSB, generally from $60 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.

This proposal also includes two vendor ATPs (see Section III.D of this preamble) and four revised EPA methods (see Section III.A of this preamble) which EPA proposes to incorporate by reference. The ATPs and EPA methods are available free of charge on their respective websites (*sgsaxys.com/wp-content/uploads/2022/09/SGS-AXYS-Method-16130-Rev-1.0.pdf, pacelabs.com* and *epa.gov/cwa-methods/approved-cwa-chemical-test-methods*), therefore the ATPs and EPA methods incorporated by reference are reasonably available.

V. Statutory and Executive Order Reviews

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

This action is not a significant regulatory action and was therefore not submitted to the Office of Management and Budget (OMB) for review.

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

The Agency certifies that this action would not have a significant economic impact on a substantial number of small entities under the Regulatory Flexibility Act. This action would 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, EPA expects the proposed revisions would lead to few, if any, increased costs. 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.,* ASTM Method D7511). Because these methods would be alternatives rather than requirements, there are no direct costs associated with this proposal. 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 of this preamble.

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 would 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. EPA does not expect the proposal would lead to any costs to any tribal governments, and if incurred, EPA 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

EPA interprets Executive Order 13045 as applying only to those regulatory actions that concern environmental health or safety risks that 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. 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 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

Executive Order 12898 (59 FR 7629, February 16, 1994) directs Federal agencies, to the greatest extent practicable and permitted by law, to make environmental justice part of their mission by identifying and addressing, as appropriate, disproportionately high and adverse human health or environmental effects of their programs, policies, and activities on minority populations (people of color) and low-income populations.

EPA believes that this type of action does not concern human health or environmental conditions and therefore cannot be evaluated with respect to potentially disproportionate and adverse effects on people of color, low-income populations and/or indigenous peoples. This action has no effect on human health or the environment because this action would approve new and revised versions of CWA testing procedures. 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. These proposed changes would 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.

List of Subjects in 40 CFR Part 136

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

Michael S. Regan,

Administrator.

For the reasons set out in the preamble, the EPA proposes to amend 40 CFR part 136 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 as follows:

■ a. Revise tables IA, IB, IC, ID, and IH in paragraph (a);

■ b. Revise the introductory text to paragraph (b) and paragraphs (b)(8)(ii) through (v), (b)(10)(i), (viii) through (xiv), (xvi) through (xxvi), (xxviii) through (xxxv), (xxxvii), (xxxix) through (li), (lv) through (lxiii), and (lxvii), (b)(15)(xi), (xx), (xxx), (xxxii), (lix), (lxv) through (lxvii), and (lxix);

■ c. Redesignate paragraphs (b)(33) through (39) as paragraphs (b)(35) through (41);

■ d. Add new paragraphs (b)(33) and (34); and

■ e. In paragraph (e), table II, revise Footnote ''5''.

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 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].		
2. Coliform (fecal), number per 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 132[3]	9221 E–2014; 9221 F–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]	B–0050–85[4].	
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	p. 108[3]	9222 B–2015[30]	B–0025–85[4].	
	MF,[2,5] two step with enrichment.	p. 111[3]	9222 B–2015[30].		
4. *E. coli*, number per 100 mL.	MPN[6,8,16] multiple tube, or.		9221 B2014/9221 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	1603.1[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.		

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

Parameter and units	Method [1]	EPA	Standard methods	AOAC, ASTM, USGS	Other
	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.	1600.1 [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.	*Water flea, Cladoceran, Ceriodaphnia dubia* acute.	2002.0 [25].			
	Water fleas, Cladocerans, Daphnia pulex and Daphnia magna acute.	2021.0 [25].			
	Fish, Fathead minnow, *Pimephales promelas*, and Bannerfin shiner, *Cyprinella leedsi*, acute.	2000.0 [25].			
	Fish, 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].			
	Fish, Sheepshead minnow, *Cyprinodon variegatus*, acute.	2004.0 [25].			
	Fish, Silverside, *Menidia beryllina, Menidia menidia,* and *Menidia peninsulae*, acute.	2006.0 [25].			
10. Toxicity, chronic, fresh water organisms, NOEC or IC_{25}, percent effluent.	Fish, Fathead minnow, *Pimephales promelas*, larval survival and growth.	1000.0 [26].			
	Fish, Fathead minnow, *Pimephales promelas*, embryo-larval survival and teratogenicity.	1001.0 [26].			
	Water flea, Cladoceran, *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.	Fish, Sheepshead minnow, *Cyprinodon variegatus*, larval survival and growth.	1004.0 [27].			

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

Parameter and units	Method[1]	EPA	Standard methods	AOAC, ASTM, USGS	Other
	Fish, Sheepshead minnow, *Cyprinodon variegatus,* embryo-larval survival and teratogenicity.	1005.0[27].			
	Fish, 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. U.S. 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 1603.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified Membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC), [in draft as of 2023]. 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 1600.1: Enterococci in Water by Membrane Filtration Using Membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), [in draft as of 2023]. 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; and U.S. EPA Whole Effluent Toxicity Methods Errata Sheet, EPA 821–R–02–012–ES. December 2016.

[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; and U.S. EPA Whole Effluent Toxicity Methods Errata Sheet, EPA 821–R–02–012–ES. December 2016.

[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; and U.S. EPA Whole Effluent Toxicity Methods Errata Sheet, EPA 821–R–02–012–ES. December 2016.

[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] 9221F. 2–2014 allows for simultaneous detection of *E. coli* and thermotolerant fecal coliforms by adding inverted vials to EC–MUG; the inverted vials collect gas produced by thermotolerant fecal 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–2020	D1067–16	I–1020–85.[2]
2. Alkalinity, as $CaCO_3$, mg/L.	Electrometric or Colorimetric titration to pH 4.5, Manual.		2320 B–2021	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–2019 or 3111 E–2019.		I–3051–85.[2]
	AA furnace		3113 B–2020.		
	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020.		
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–2021		973.49.[3]
	Nesslerization			D1426–15 (A)	973.49,[3] I–3520–85.[2]
	Titration		4500–NH_3 C–2021.		
	Electrode		4500–NH_3 D–2021 or E–2021.	D1426–15 (B).	
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction-based methods.		4500–NH_3 F–2021		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–2021 4500–NH_3 H–2021.		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–2019.		
	AA furnace		3113 B–2020.		
	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–2020	D1976–20.	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020 or 3114 C–2020.	D2972–15 (B)	I–3062–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14,[3] I–4020–05.[70]
	Colorimetric (SDDC)		3500–As B–2020	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–2019		I–3084–85.[2]
	AA furnace		3113 B–2020	D4382–18.	
	ICP/AES[36]	200.5, Rev 4.2 (2003);[68] 200.7, Rev. 4.4 (1994).	3120 B–2020		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2019 or 3111 E–2019.	D3645–15 (A)	I–3095–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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[85]		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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020, C–2020 or D–2020.	D4327–17	993.30,[3] I–2057–85.[79]
	CIE/UV		4140 B–2020	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–2019 or 3111 C–2019.	D3557–17 (A or B)	974.27,[3] p. 37,[9] I–3135–85[2] or I–3136–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–1472–85[2] or I–4471–97.[50]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14,[3] I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Voltammetry[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–2019 or 3111 D–2019.	D511–14 (B)	I–3152–85.[2]
	ICP/AES	200.5, Rev 4.2 (2003);[68] 200.7, Rev. 4.4 (1994).	3120 B–2020		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14.[3]
	DCP				See footnote.[34]
	Titrimetric (EDTA)		3500–Ca B–2020	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[85]		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–2021	D512–12 (B)	I–1183–85.[2]
	(Mercuric nitrate)		4500–Cl$^-$ C–2021	D512–12 (A)	973.51,[3] I–1184–85.[2]
	Colorimetric: manual				I–1187–85.[2]
	Automated (ferricyanide).		4500–Cl$^-$ E–2021		I–2187–85.[2]
	Potentiometric Titration.		4500–Cl$^-$ D–2021.		
	Ion Selective Electrode.			D512–12 (C).	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B–2020 or 4110 C–2020.	D4327–17	993.30,[3] I–2057–90.[51]
	CIE/UV		4140 B–2020	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–2019		I–1232–85.[2]
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500–Cr C–2020	D5257–17	993.23.[3]
	Colorimetric (diphenyl-carbazide).		3500–Cr B–2020	D1687–17 (A)	I–1230–85.[2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
19. Chromium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration[36]		3111 B–2019	D1687–17 (B)	974.27,[3] I–3236–85.[2]
	AA chelation-extraction.		3111 C–2019.		
	AA furnace		3113 B–2020	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–2020	D1976–20.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020.		
20. Cobalt—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 B–2019 or 3111 C–2019.	D3558–15 (A or B)	p. 37,[9] I–3239–85.[2]
	AA furnace		3113 B–2020	D3558–15 (C)	I–4243–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2021[78].		
	Platinum cobalt visual comparison.		2120 B–2021		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–2019 or 3111 C–2019.	D1688–17 (A or B)	974.27,[3] p. 37,[9] I–3270–85[2] or I–3271–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020.		
	Colorimetric (Bathocuproine).		3500–Cu C–2020		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.		4500–CN^- P–2021	D7511–12(17).	
	Manual distillation with $MgCl_2$, followed by any of the following:	335.4, Rev. 1.0 (1993)[57].	4500–CN^- B–2021 and C–2021.	D2036–09(15)(A), D7284–20.	10–204–00–1–X.[56]
	Flow Injection, gas diffusion amperometry.			D2036–09(15)(A) D7284–20.	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	Titrimetric		4500–CN^- D–2021	D2036–09(15)(A)	p. 22.[9]
	Spectrophotometric, manual.		4500–CN^- E–2021	D2036–09(15)(A)	I–3300–85.[2]
	Semi-Automated[20]	335.4, Rev. 1.0 (1993)[57].	4500–CN^- N–2021		10–204–00–1–X,[56] I–4302–85.[2]
	Ion Chromatography			D2036–09(15)(A).	
	Ion Selective Electrode.		4500–CN^- F–2021	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–2021	D2036–09(15)(B).	
	Flow injection and ligand exchange, followed by gas diffusion amperometry[59].		4500–CN^- Q–2021	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.		4500–CN^- R–2021	D7237–18 (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–2021	D1179–16 (A).	
	Electrode, manual		4500–F^- C–2021	D1179–16 (B).	
	Electrode, automated		4500–F^- G–2021		I–4327–85.[2]
	Colorimetric, (SPADNS).		4500–F^- D–2021.		
	Automated complexone.		4500–F^- E–2021.		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B–2020 or C–2020.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2020	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–2019.		
	AA furnace	231.2 (Issued 1978)[1]	3113 B–2020.		
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2021	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–2021.		
28. Hydrogen ion (pH), pH units.	Electrometric measurement.		4500–H^+ B–2021	D1293–18 (A or B)	973.41,[3] I–1586–85.[2]
	Automated electrode	150.2 (Dec. 1982)[1]			See footnote,[21] I–2587–85.[2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
29. Iridium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 B–2019.		
	AA furnace	235.2 (Issued 1978)[1].			
	ICP/MS		3125 B–2020.		
30. Iron—Total,[4] mg/L	Digestion,[4] followed by any of the following:				
	AA direct aspiration[36]		3111 B–2019 or 3111 C–2019.	D1068–15 (A)	974.27,[3] I–3381–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2021 or C–2021 and 4500–NH_3 B–2021.	D3590–17 (A)	I–4515–91.[45]
	Titration		4500–NH_3 C–2021		973.48.[3]
	Nesslerization			D1426–15 (A).	
	Electrode		4500–NH_3 D–2021 or E–2021.	D1426–15 (B).	
	Semi-automated phenate.	350.1, Rev. 2.0 (1993).	4500–NH_3 G–2021 or 4500–NH_3 H–2021.		
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.		4500–NH_3 F–2021		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–2021	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]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	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–2019 or 3111 C–2019.	D3559–15 (A or B)	974.27,[3] I–3399–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14,[3] I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Voltammetry[11]			D3559–15 (C).	
	Colorimetric (Dithizone).		3500-Pb B–2020.		
33. Magnesium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 B–2019	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14.[3]
	DCP				See footnote.[34]
	Ion Chromatography			D6919–17.	
34. Manganese—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration[36]		3111 B–2019 or 3111 C–2019.	D858–17 (A or B)	974.27,[3] I–3454–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14,[3] I–4472–97.[81]
	DCP[36]			D4190–15	See footnote.[34]
	Colorimetric (Persulfate).		3500–Mn B–2020		920.203.[3]
	Colorimetric (Periodate).				See footnote.[23]
35. Mercury—Total, mg/L.	Cold vapor, Manual	245.1, Rev. 3.0 (1994).	3112 B–2020	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–2019		I–3490–85.[2]
	AA furnace		3113 B–2020		I–3492–96.[47]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2020	D1976–20	I–4471–97.[50]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2019 or 3111 C–2019.	D1886–14 (A or B)	I–3499–85.[2]
	AA furnace		3113 B–2020	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020 or C–2020.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2020	D6508–15	D6508, Rev. 2.[54]
	Ion Selective Electrode.		4500–NO_3^- D–2019.		
	Colorimetric (Brucine sulfate).	352.1 (Issued 1971)[1]			973.50,[3] 419D,[86] 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–2019	D3867–16 (B).	
	Cadmium reduction, Automated.	353.2, Rev. 2.0 (1993).	4500–NO_3^- F–2019 or 4500–NO_3^- I–2019.	D3867–16 (A)	I–2545–90.[51]
	Automated hydrazine		4500–NO_3^- H–2019.		
	Reduction/Colorimetric.				See footnote.[62]
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2020 or C–2020.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2020	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–2021		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–2019 4500–NO_3^- I–2019.	D3867–16 (A)	I–4545–85.[2]
	Manual (*bypass cadmium or enzymatic reduction).		4500–NO_3^- E–2019, 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–2020 or C–2020.	D4327–17	993.30.[3]
	CIE/UV		4140 B–2020	D6508–15	D6508, Rev. 2.[54]
	Automated (*bypass Enzymatic reduction).			D7781–14	I–2547–11,[72] I–2548–11,[72] N07–0003.[73]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
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 or G–2021[38].		
	Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry.	1664 Rev. A; 1664 Rev. B[42].	5520 B or G–2021[38] and 5520 F–2021[38].		
42. Organic carbon—Total (TOC), mg/L.	Combustion	....	5310 B–2014	D7573–18a^{e1}	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–2021 or G–2021.	....	973.56,[3] I–4601–85,[2] I–2601–90.[80]
	Manual, single-reagent.	....	4500–P E–2021	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–2020 or C–2020.	D4327–17	993.30.[3]
	CIE/UV	....	4140 B–2020	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–2019.		
	AA furnace	252.2 (Issued 1978)[1].			
46. Oxygen, dissolved, mg/L.	Winkler (Azide modification).	....	4500–O (B–F)-2021	D888–18 (A)	973.45B,[3] I–1575–78.[8]
	Electrode	....	4500–O G–2021	D888–18 (B)	I–1576–78.[8]
	Luminescence-Based Sensor.	....	4500–O H–2021	D888–18 (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–2019.		
	AA furnace	253.2 (Issued 1978)[1].			
	ICP/MS	....	3125 B–2020.		
	DCP	....	....	....	See footnote.[34]
48. Phenols, mg/L	Manual distillation,[26] followed by any of the following:.	420.1 (Rev. 1978)[1]	5530 B–2021	D1783–01(12).	
	Colorimetric (4AAP) manual.	420.1 (Rev. 1978)[1]	5530 D–2021[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)–2021	....	973.55.[3]
	Manual	365.3 (Issued 1978)[1]	4500–P E–2021	D515–88 (A).	
	Automated ascorbic acid reduction.	365.1 Rev. 2.0 (1993)	4500–P (F–H)–2021	....	973.56,[3] I–4600–85.[2]
	ICP/AES[4][36]	200.7, Rev. 4.4 (1994).	3120 B–2020	....	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–2019.		

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	AA furnace	255.2 (Issued 1978)[1].			
	ICP/MS		3125 B–2020.		
	DCP				See footnote.[34]
52. Potassium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 B–2019		973.53,[3] I–3630–85.[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2020.		
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14.[3]
	Flame photometric		3500–K B–2020.		
	Electrode		3500–K C–2020.		
	Ion Chromatography			D6919–17.	
53. Residue—Total, mg/L.	Gravimetric, 103–105°.		2540 B–2020		I–3750–85.[2]
54. Residue—filterable, mg/L.	Gravimetric, 180°		2540 C–2020	D5907–18 (B)	I–1750–85.[2]
55. Residue—non-filterable (TSS), mg/L.	Gravimetric, 103–105° post-washing of residue.		2540 D–2020	D5907–18 (A)	I–3765–85.[2]
56. Residue—settleable, mg/L.	Volumetric (Imhoff cone), or gravimetric.		2540 F–2020.		
57. Residue—Volatile, mg/L.	Gravimetric, 550°	160.4 (Issued 1971)[1]	2540 E–2020		I–3753–85.[2]
58. Rhodium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration, or.		3111 B–2019.		
	AA furnace	265.2 (Issued 1978)[1].			
	ICP/MS		3125 B–2020.		
59. Ruthenium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration, or.		3111 B–2019.		
	AA furnace	267.2[1].			
	ICP/MS		3125 B–2020.		
60. Selenium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA furnace		3113 B–2020	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–2020	D1976–20.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14,[3] I–4020–05,[70] I–4472–97.[81]
	AA gaseous hydride		3114 B–2020, or 3114 C–2020.	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–2021	D859–16	I–1700–85.[2]
	Automated (Molybdosilicate).		4500–SiO_2 E–2021 or F–2021.		I–2700–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003);[68] 200.7, Rev. 4.4 (1994).	3120 B–2020		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2019 or 3111 C–2019.		974.27,[3] p. 37,[9] I–3720–85.[2]
	AA furnace		3113 B–2020		I–4724–89.[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	200.5, Rev. 4.2 (2003);[68] 200.7, Rev. 4.4 (1994).	3120 B–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2019		973.54,[3] I–3735–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003);[68] 200.7, Rev. 4.4 (1994).	3120 B–2020		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14.[3]
	DCP				See footnote.[34]
	Flame photometric		3500-Na B–2020.		
	Ion Chromatography			D6919–17.	
64. Specific conductance, micromhos/cm at 25 °C.	Wheatstone bridge	120.1 (Rev. 1982)[1]	2510 B–2021	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–2021 or G–2021.		
	Gravimetric		4500–SO_4^{2-} C–2021 or D–2021.		925.54.[3]
	Turbidimetric		4500–SO_4^{2-} E–2021	D516–16.	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2020 or C–2020.	D4327–17	993.30,[3] I–4020–05.[70]
	CIE/UV		4140 B–2020	D6508–15	D6508, Rev. 2.[54]
66. Sulfide (as S), mg/L.	Sample Pretreatment		4500–S^{2-} B, C–2021.		
	Titrimetric (iodine)		4500–S^{2-} F–2021		I–3840–85.[2]
	Colorimetric (methylene blue).		4500–S^{2-} D–2021.		
	Ion Selective Electrode.		4500–S^{2-} G–2021	D4658–15.	
67. Sulfite (as SO_3), mg/L.	Titrimetric (iodine-iodate).		4500–SO_3^{2-} B–2021.		
68. Surfactants, mg/L	Colorimetric (methylene blue).		5540 C–2021	D2330–20.	
69. Temperature, °C	Thermometric		2550 B–2010		See footnote.[32]
70. Thallium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 B–2019.		
	AA furnace	279.2 (Issued 1978)[1]	3113 B–2020.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B–2020	D1976–20.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2019		I–3850–78.[8]
	AA furnace		3113 B–2020.		
	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–2020	D5673–16	993.14.[3]
72. Titanium—Total,[4] mg/L.	Digestion,[4] followed by any of the following:				
	AA direct aspiration		3111 D–2019.		
	AA furnace	283.2 (Issued 1978)[1].			
	ICP/AES	200.7, Rev. 4.4 (1994).			

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods[84]	ASTM	USGS/AOAC/other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	D5673–16	993.14.[3]
	DCP				See footnote.[34]
73. Turbidity, NTU[53]	Nephelometric	180.1, Rev. 2.0 (1993).	2130 B–2020	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–2019.		
	AA furnace		3113 B–2020	D3373–17.	
	ICP/AES	200.5, Rev. 4.2 (2003);[68] 200.7, Rev. 4.4 (1994).	3120 B–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2019 or 3111 C–2019.	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–2020	D1976–20	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2020	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–2020		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 of this section); 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, 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 of this section for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).

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

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

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

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

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

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

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

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

[72] 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 2018, MACHEREY-NAGEL GmbH and Co. KG.

[84] Please refer to the following applicable Quality Control Sections: Part 2000 Methods, Physical and Aggregate Properties 2020 (2021); Part 3000 Methods, Metals, 3020 (2021); Part 4000 Methods, Inorganic Nonmetallic Constituents, 4020 (2022); Part 5000 Methods, and Aggregate Organic Constituents, 5020 (2022). These Quality Control Standards are available for download at *www.standardmethods.org* at no charge.

[85] Each laboratory may establish its own control limits by performing at least 25 glucose-glutamic acid (GGA) checks over several weeks or months and calculating the mean and standard deviation. The laboratory may then use the mean ± 3 standard deviations as the control limit for future GGA checks. However, GGA acceptance criteria can be no wider than 198 ± 30.5 mg/L for BOD$_5$. GGA acceptance criteria for CBOD must be either 198 ± 30.5 mg/L, or the lab may develop control charts under the following conditions: dissolved oxygen uptake from the seed contribution is between 0.6–1.0 mg/L; control charts are performed on at least 25 GGA checks with three standard deviations from the derived mean; the RSD must not exceed 7.5%; and any single GGA value cannot be less than 150 mg/L or higher than 250 mg/L.

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

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

Parameter[1]	Method	EPA[2 7]	Standard methods[15]	ASTM	Other
1. Acenaphthene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
2. Acenaphthylene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	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–2020		See footnote,[9] p. 27.
	HPLC	610	6440B–2021	D4657–92 (98).	
6. Benzene	GC	602	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		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–2020.		
	HPLC	605.			
8. Benzo(a)anthracene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
9. Benzo(a)pyrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
10. Benzo(b)fluoranthene.	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
11. Benzo(g,h,i)perylene.	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
12. Benzo(k)fluoranthene.	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
13. Benzyl chloride	GC				See footnote,[3] p. 130.
	GC/MS				See footnote,[6] p. S102.
14. Butyl benzyl phthalate.	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] 27.
15. bis(2-Chloroethoxy) methane.	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
16. bis(2-Chloroethyl) ether.	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
17. bis(2-Ethylhexyl) phthalate.	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
18. Bromodichloromethane.	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
19. Bromoform	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
20. Bromomethane	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
21. 4-Bromophenyl phenyl ether.	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
22. Carbon tetrachloride.	GC	601	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]

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

Parameter[1]	Method	EPA[2] [7]	Standard methods[15]	ASTM	Other
23. 4-Chloro-3-methyl phenol.	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
24. Chlorobenzene	GC	601, 602	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
25. Chloroethane	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96.[13]
26. 2-Chloroethylvinyl ether.	GC	601.			
	GC/MS	624.1, 1624B.			
27. Chloroform	GC	601	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
28. Chloromethane	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
29. 2-Chloronaphthalene.	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
30. 2-Chlorophenol	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
31. 4-Chlorophenyl phenyl ether.	GC	611.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
32. Chrysene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
33. Dibenzo(a,h)anthracene.	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
34. Dibromochloromethane.	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
35. 1,2-Dichlorobenzene.	GC	601, 602	6200 C–2020.		
	GC/MS	624.1, 1625B	6200 B–2020		See footnote,[9] p. 27 O–4127–96,[13] O–4436–16.[14]
36. 1,3-Dichlorobenzene.	GC	601, 602	6200 C–2020.		
	GC/MS	624.1, 1625B	6200 B–2020		See footnote,[9] p. 27 O–4127–96.[13]
37. 1,4-Dichlorobenzene.	GC	601, 602	6200 C–2020.		
	GC/MS	624.1, 1625B	6200 B–2020		See footnote,[9] p. 27 O–4127–96,[13] O–4436–16.[14]
38. 3,3′-Dichlorobenzidine.	GC/MS	625.1, 1625B	6410 B–2020.		
	HPLC	605.			
39. Dichlorodifluoromethane.	GC	601.			
	GC/MS		6200 B–2020		O–4127–96,[13] O–4436–16.[14]
40. 1,1-Dichloroethane	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
41. 1,2-Dichloroethane	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
42. 1,1-Dichloroethene	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
43. *trans*-1,2-Dichloroethene.	GC	601	6200 C–2020.		

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

Parameter [1]	Method	EPA [2] [7]	Standard methods [15]	ASTM	Other
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
44. 2,4-Dichlorophenol	GC	604	6420 B–2020..		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
45. 1,2-Dichloropropane.	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
46. *cis*-1,3-Dichloropropene.	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
47. *trans*-1,3-Dichloropropene.	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
48. Diethyl phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
49. 2,4-Dimethylphenol	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
50. Dimethyl phthalate	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
51. Di-*n*-butyl phthalate.	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
52. Di-*n*-octyl phthalate.	GC	606.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
53. 2, 4-Dinitrophenol	GC	604	6420 B–2020		See footnote,[9] p. 27.
	GC/MS	625.1, 1625B	6410 B–2020.		
54. 2,4-Dinitrotoluene	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
55. 2,6-Dinitrotoluene	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2020		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–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
58. Fluoranthene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
59. Fluorene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran.	GC/MS	1613B [10]			ATM 16130,[15] PAM 16130–SSI.[16]
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran.	GC/MS	1613B [10]			ATM 16130,[15] PAM 16130–SSI.[16]
62. 1,2,3,4,6,7,8-Heptachloro-dibenzo-*p*-dioxin.	GC/MS	1613B [10]			ATM 16130,[15] PAM 16130–SSI.[16]
63. Hexachlorobenzene.	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
64. Hexachlorobutadiene.	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27 O–4127–96.[13]
65. Hexachlorocyclopentadiene.	GC	612.			
	GC/MS	625.1,[5] 1625B	6410 B–2020		See footnote,[9] p. 27 O–4127–96.[13]
66. 1,2,3,4,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B [10]			ATM 16130,[15] PAM 16130–SSI.[16]

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

Parameter[1]	Method	EPA[2][7]	Standard methods[15]	ASTM	Other
67. 1,2,3,6,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
68. 1,2,3,7,8,9-Hexachloro-dibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
69. 2,3,4,6,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
70. 1,2,3,4,7,8-Hexachloro-dibenzo-*p*-dioxin.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI,[16] G BHTYHGTGB B VB B5 BV.
71. 1,2,3,6,7,8-Hexachloro-dibenzo-*p*-dioxin.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
72. 1,2,3,7,8,9-Hexachloro-dibenzo-*p*-dioxin.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
73. Hexachloroethane	GC	612.			
	GC/MS	625.1, 1625B	6410 B–2020		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–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
75. Isophorone	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
76. Methylene chloride	GC	601	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
77. 2-Methyl-4,6-dinitrophenol.	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
78. Naphthalene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021.		
79. Nitrobenzene	GC	609.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC			D4657–92 (98).	
80. 2-Nitrophenol	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
81. 4-Nitrophenol	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
82. N-Nitrosodimethylamine.	GC	607.			
	GC/MS	625.1,[5] 1625B	6410 B–2020		See footnote,[9] p. 27.
83. N-Nitrosodi-*n*-propylamine.	GC	607.			
	GC/MS	625.1,[5] 1625B	6410 B–2020		See footnote,[9] p. 27.
84. N-Nitrosodiphenylamine.	GC	607.			
	GC/MS	625.1,[5] 1625B	6410 B–2020		See footnote,[9] p. 27.
85. Octachlorodibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
86. Octachlorodibenzo-*p*-dioxin.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
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–2020		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–2020.		

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

Parameter[1]	Method	EPA[2][7]	Standard methods[15]	ASTM	Other
89. PCB–1221	GC	608.3			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2020.		
90. PCB–1232	GC	608.3			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2020.		
91. PCB–1242	GC	608.3			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2020.		
92. PCB–1248	GC	608.3			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2020.		
93. PCB–1254	GC	608.3			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2020.		
94. PCB–1260	GC	608.3			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625.1	6410 B–2020.		
95. 1,2,3,7,8-Pentachloro-dibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
96. 2,3,4,7,8-Pentachloro-dibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
97. 1,2,3,7,8-Pentachloro-dibenzo-*p*-dioxin.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
98. Pentachlorophenol	GC	604	6420 B–2020		See footnote,[3] p. 140.
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
99. Phenanthrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
100. Phenol	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
101. Pyrene	GC	610.			
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
	HPLC	610	6440 B–2021	D4657–92 (98).	
102. 2,3,7,8-Tetra-chloro-dibenzofuran.	GC/MS	1613B[10]			ATM 16130,[15] PAM 16130–SSI.[16]
103. 2,3,7,8-Tetra-chloro-dibenzo-*p*-dioxin.	GC/MS	613, 625.1,[5a] 1613B			ATM 16130,[15] PAM 16130–SSI.[16]
104. 1,1,2,2-Tetrachloroethane.	GC	601	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96.[13]
105. Tetrachloroethene.	GC	601	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
106. Toluene	GC	602	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		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–2020		See footnote,[9] p. 27 O–4127–96,[13] O–4436–16.[14]
108. 1,1,1-Trichloro-ethane.	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
109. 1,1,2-Trichloro-ethane.	GC	601	6200 C–2020		See footnote,[3] p. 130.
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
110. Trichloroethene	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		O–4127–96,[13] O–4436–16.[14]
111. Trichlorofluorometh-ane.	GC	601	6200 C–2020.		

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

Parameter[1]	Method	EPA[2] [7]	Standard methods[15]	ASTM	Other
	GC/MS	624.1	6200 B–2020		O–4127–96.[13]
112. 2,4,6-Trichlorophenol.	GC	604	6420 B–2020.		
	GC/MS	625.1, 1625B	6410 B–2020		See footnote,[9] p. 27.
113. Vinyl chloride	GC	601	6200 C–2020.		
	GC/MS	624.1, 1624B	6200 B–2020		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.

[15] Please refer to the following Quality Control Section: Part 6000 Individual Organic Compounds, 6020 (2019)[16] SGS AXYS Method ATM 16130, "Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs) Using Waters and Agilent Gas Chromatography-Tandem-Mass Spectrometry (GC/MS/MS), Revision 1.0, " is available at: *https://www.sgsaxys.com/wp-content/uploads/2022/09/SGS-AXYS-Method-16130-Rev-1.0.pdf.*

[16] Pace Analytical Method PAM–16130–SSI, "Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs) Using Shimadzu Gas Chromatography Mass Spectrometry (GC–MS/MS), Revision 1.1," is available at: *www.pacelabs.com.*

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

Parameter	Method	EPA [2] [7] [10]	Standard methods [15]	ASTM	Other
1. Aldrin	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96 (02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
2. Ametryn	GC	507, 619			See footnote,[3] p. 83; See footnote,[9] O–3106–93; See footnote,[6] p. S68.
	GC/MS	525.2, 625.1			See footnote,[14] O–1121–91.
3. Aminocarb	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
4. Atraton	GC	619			See footnote,[3] p. 83; See footnote,[6] p. S68.
	GC/MS	625.1.			
5. Atrazine	GC	507, 619, 608.3			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	HPLC/MS				See footnote,[12] O–2060–01.
	GC/MS	525.1, 525.2, 625.1			See footnote,[11] O–1126–95.
6. Azinphos methyl	GC	614, 622, 1657			See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC–MS	625.1			See footnote,[11] O–1126–95.
7. Barban	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	GC/MS	625.1.			
8. α-BHC	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[8] 3M0222.
	GC/MS	625.1 [5]	6410 B–2020		See footnote,[11] O–1126–95.
9. β-BHC	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
10. δ-BHC	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
11. γ-BHC (Lindane)	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625.1 [5]	6410 B–2020		See footnote,[11] O–1126–95.
12. Captan	GC	617, 608.3	6630 B–2021	D3086–90, D5812–96(02).	See footnote,[3] p. 7.
13. Carbaryl	TLC				See footnote,[3] p. 94, See footnote,[6] p. S60.
	HPLC	531.1, 632.			
	HPLC/MS	553			See footnote,[12] O–2060–01.
	GC/MS	625.1			See footnote,[11] O–1126–95.
14. Carbophenothion	GC	617, 608.3	6630 B–2021		See footnote,[4] page 27; See footnote,[6] p. S73.
	GC/MS	625.1.			

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

Parameter	Method	EPA [2] [7] [10]	Standard methods [15]	ASTM	Other
15. Chlordane	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
16. Chloropropham	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	GC/MS	625.1.			
17. 2,4–D	GC	615	6640 B–2021		See footnote,[3] p. 115; See footnote,[4] O–3105–83.
	HPLC/MS				See footnote,[12] O–2060–01.
18. 4,4′-DDD	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3105–83; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
19. 4,4′-DDE	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020		See footnote,[11] O–1126–95.
20. 4,4′-DDT	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
21. Demeton-O	GC	614, 622			See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC/MS	625.1.			
22. Demeton-S	GC	614, 622			See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC/MS	625.1.			
23. Diazinon	GC	507, 614, 622, 1657			See footnote,[3] p. 25; See footnote,[4] O–3104–83; See footnote,[6] p. S51.
	GC/MS	525.2, 625.1			See footnote,[11] O–1126–95.
24. Dicamba	GC	615			See footnote,[3] p. 115.
	HPLC/MS				See footnote,[12] O–2060–01.
25. Dichlofenthion	GC	622.1			See footnote,[4] page 27; See footnote,[6] p. S73.
26. Dichloran	GC	608.2, 617, 608.3	6630 B–2021		See footnote,[3] p. 7.
27. Dicofol	GC	617, 608.3			See footnote,[4] O–3104–83.
28. Dieldrin	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020		See footnote,[11] O–1126–95.
29. Dioxathion	GC	614.1, 1657			See footnote,[4] page 27; See footnote,[6] p. S73.
30. Disulfoton	GC	507, 614, 622, 1657			See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC/MS	525.2, 625.1			See footnote,[11] O–1126–95.
31. Diuron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.

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

Parameter	Method	EPA[2][7][10]	Standard methods[15]	ASTM	Other
	HPLC	632.			
	HPLC/MS	553			See footnote,[12] O–2060–01.
32. Endosulfan I	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M022).
	GC/MS	625.1[5]	6410 B–2020		See footnote,[13] O–2002–01.
33. Endosulfan II	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[8] 3M0222.
	GC/MS	625.1[5]	6410 B–2020		See footnote,[13] O–2002–01.
34. Endosulfan Sulfate	GC	617, 608.3	6630 C–2021		See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
35. Endrin	GC	505, 508, 617, 1656, 608.3.	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	525.1, 525.2, 625.1[5]	6410 B–2020.		
36. Endrin aldehyde	GC	617, 608.3	6630 C–2021		See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
37. Ethion	GC	614, 614.1, 1657			See footnote,[4] page 27; See footnote,[6] p. S73.
	GC/MS	625.1			See footnote,[13] O–2002–01.
38. Fenuron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
39. Fenuron-TCA	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
40. Heptachlor	GC	505, 508, 617, 1656, 608.3.	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	525.1, 525.2, 625.1	6410 B–2020.		
41. Heptachlor epoxide.	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[6] p. S73; See footnote,[8] 3M0222.
	GC/MS	625.1	6410 B–2020.		
42. Isodrin	GC	617, 608.3	6630 B–2021 & C–2021.		See footnote,[4] O–3104–83; See footnote,[6] p. S73.
	GC/MS	625.1.			
43. Linuron	GC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS	553			See footnote,[12] O–2060–01.
	GC/MS				See footnote,[11] O–1126–95.
44. Malathion	GC	614, 1657	6630 B–2021		See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC/MS	625.1			See footnote,[11] O–1126–95.
45. Methiocarb	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.

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

Parameter	Method	EPA[2 7 10]	Standard methods[15]	ASTM	Other
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
46. Methoxychlor	GC	505, 508, 608.2, 617, 1656, 608.3.	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	525.1, 525.2, 625.1			See footnote,[11] O–1126–95.
47. Mexacarbate	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
	GC/MS	625.1.			
48. Mirex	GC	617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83.
	GC/MS	625.1.			
49. Monuron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
50. Monuron-TCA	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
51. Neburon	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
52. Parathion methyl	GC	614, 622, 1657	6630 B–2021		See footnote,[4] page 27; See footnote,[3] p. 25.
	GC/MS	625.1			See footnote,[11] O–1126–95.
53. Parathion ethyl	GC	614	6630 B–2021		See footnote,[4] page 27; See footnote,[3] p. 25.
	GC/MS				See footnote,[11] O–1126–95.
54. PCNB	GC	608.1, 617, 608.3	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7.
55. Perthane	GC	617, 608.3		D3086–90, D5812–96(02).	See footnote,[4] O–3104–83.
56. Prometon	GC	507, 619			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.2, 625.1			See footnote,[11] O–1126–95.
57. Prometryn	GC	507, 619			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2, 625.1			See footnote,[13] O–2002–01.
58. Propazine	GC	507, 619, 1656, 608.3			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2, 625.1.			
59. Propham	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.

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

Parameter	Method	EPA[2 7 10]	Standard methods[15]	ASTM	Other
60. Propoxur	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
61. Secbumeton	TLC				See footnote,[3] p. 83; See footnote,[6] p. S68.
	GC	619.			
62. Siduron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
63. Simazine	GC	505, 507, 619, 1656, 608.3.			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2, 625.1			See footnote,[11] O–1126–95.
64. Strobane	GC	617, 608.3	6630 B–2021 & C–2021.		See footnote,[3] p. 7.
65. Swep	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
66. 2,4,5–T	GC	615	6640 B–2021		See footnote,[3] p. 115; See footnote,[4] O–3105–83.
67. 2,4,5–TP (Silvex)	GC	615	6640 B–2021		See footnote,[3] p. 115; See footnote,[4] O–3105–83.
68. Terbuthylazine	GC	619, 1656, 608.3			See footnote,[3] p. 83; See footnote,[6] p. S68.
	GC/MS				See footnote,[13] O–2002–01.
69. Toxaphene	GC	505, 508, 617, 1656, 608.3.	6630 B–2021 & C–2021.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote;[8] See footnote,[4] O–3105–83.
	GC/MS	525.1, 525.2, 625.1	6410 B–2020.		
70. Trifluralin	GC	508, 617, 627, 1656, 608.3.	6630 B–2021		See footnote,[3] p. 7; See footnote,[9] O–3106–93.
	GC/MS	525.2, 625.1			See footnote,[11] O–1126–95.

Table ID notes:

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

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

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

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

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

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

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608.3 and 625.1 in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608.3 or 5% of all samples analyzed with Method 625.1 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

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

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

[10] EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, EPA 821–R–92–002, April 1992, U.S. EPA. EPA Methods 505, 507, 508, 525.1, 531.1 and 553 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821–R–93–010B, 1993, U.S. EPA. EPA Method 525.2 is in Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, Revision 2.0, 1995, U.S. EPA. EPA methods 1656 and 1657 are in Methods for The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821–R–93–010A, 1993, U.S. EPA. Methods 608.3 and 625.1 are available at: *cwa-methods/approved-cwa-test-methods-organic-compounds.*

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

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

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

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

[15] Please refer to the following applicable Quality Control Section: Part 6000 Methods, Individual Organic Compounds 6020 (2019). These Quality Control Standards are available for download at *www.standardmethods.org* at no charge.

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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–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.–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	1103.2[18]	9222 B–2015/9222 I–2015,[17] 9213 D–2007.	D5392–93.[8]	
	Single step	1603.1,[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	1106.2[22]	9230 C–2013[30]	D5259–92.[8]	
	Single step, or	1600.1[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 1103.2: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC), [in draft as of 2023]. US EPA.

[19] Method 1603.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC), [in draft as of 2023]. 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 1106.2: Enterococci in Water by Membrane Filtration Using membrane-*Enterococcus*-Esculin Iron Agar (mE–EIA), [in draft as of 2023]. US EPA.

[23] Method 1600.1: Enterococci in Water by Membrane Filtration Using membrane-*Enterococcus* Indoxyl-β-D-Glucoside Agar (mEI), [in draft as of 2023]. 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 allows for simultaneous detection of *E. coli* and thermotolerant fecal coliforms by adding inverted vials to EC–MUG; the inverted vials collect gas produced by thermotolerant fecal coliforms.

(b) The material listed in this paragraph (b) 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 is available for inspection at the EPA and at the National Archives and Records Administration (NARA). Contact the EPA at: EPA's Water Docket, EPA West, 1301 Constitution Avenue NW, Room 3334, Washington, DC 20004; telephone: 202–566–2426; email: *docket-customerservice@epa.gov.* For information on the availability of this material at NARA, visit *www.archives.gov/federal-register/cfr/ibr-locations.html* or email *fr.inspection@nara.gov.* The material may be obtained from the following sources in this paragraph (b).

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(ii) Method 1103.2: *Escherichia coli (E. coli)* in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). [in draft as of 2023]. EPA Table IH, Note 18.

(iii) Method 1106.2: Enterococci in Water by Membrane Filtration Using membrane-*Enterococcus*-Esculin Iron Agar (mE–EIA). [in draft as of 2023]. Table IH, Note 22.

(iv) Method 1600.1: Enterococci in Water by Membrane Filtration Using membrane-*Enterococcus* Indoxyl-β-D-Glucoside Agar (mEI). [in draft as of 2023]. EPA. Table 1A, Note 24; Table IH, Note 23.

(v) Method 1603.1: *Escherichia coli (E. coli)* in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). [in draft as of 2023]. EPA. Table IA, Note 21; Table IH, Note 19.

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(10) * * *

(i) *Standard Methods for the Examination of Water and Wastewater.* 14th Edition, 1975. Table IB, Notes 27 and 86.

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(viii) 2120, Color. 2021. Table IB.

(ix) 2130, Turbidity. 2020. Table IB.

(x) 2310, Acidity. 2020. Table IB.

(xi) 2320, Alkalinity. 2021. Table IB.

(xii) 2340, Hardness. 2021. Table IB.

(xiii) 2510, Conductivity. 2021. Table IB.

(xiv) 2540, Solids. 2020. Table IB.

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(xvi) 3111, Metals by Flame Atomic Absorption Spectrometry. 2019. Table IB.

(xvii) 3112, Metals by Cold-Vapor Atomic Absorption Spectrometry. 2020. Table IB.

(xviii) 3113, Metals by Electrothermal Atomic Absorption Spectrometry. 2020. Table IB.

(xix) 3114, Arsenic and Selenium by Hydride Generation/Atomic Absorption Spectrometry. 2020, Table IB.

(xx) 3120, Metals by Plasma Emission Spectroscopy. 2020. Table IB.

(xxi) 3125, Metals by Inductively Coupled Plasma-Mass Spectrometry. 2020. Table IB.

(xxii) 3500–Al, Aluminum. 2020. Table IB.

(xxiii) 3500–As, Arsenic. 2020. Table IB.

(xxiv) 3500–Ca, Calcium. 2020. Table IB.

(xxv) 3500–Cr, Chromium. 2020. Table IB.

(xxvi) 3500–Cu, Copper. 2020. Table IB.

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(xxviii) 3500–Pb, Lead. 2020. Table IB.

(xxix) 3500–Mn, Manganese. 2020. Table IB.

(xxx) 3500–K, Potassium. 2020. Table IB.

(xxxi) 3500–Na, Sodium. 2020. Table IB.

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(xxxiii) 3500–Zn, Zinc. 2020. Table IB.

(xxxiv) 4110, Determination of Anions by Ion Chromatography. 2020. Table IB.

(xxxv) 4140, Inorganic Anions by Capillary Ion Electrophoresis. 2020. Table IB.

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(xxxvii) 4500 Cl^-, Chloride. 2021. Table IB.

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(xxxix) 4500–CN^-, Cyanide. 2021. Table IB.

(xl) 4500–F^-, Fluoride. 2021. Table IB.

(xli) 4500–H^+, pH Value. 2021. Table IB.

(xlii) 4500–NH_3, Nitrogen (Ammonia). 2021. Table IB.

(xliii) 4500–NO_2^-, Nitrogen (Nitrite). 2021. Table IB.

(xliv) 4500–NO_3^-, Nitrogen (Nitrate). 2019. Table IB.

(xlv) 4500–$N_{(org)}$, Nitrogen (Organic). 2021. Table IB.

(xlvi) 4500–O, Oxygen (Dissolved). 2021. Table IB.

(xlvii) 4500–P, Phosphorus. 2021. Table IB.

(xlviii) 4500–SiO_2, Silica. 2021. Table IB.

(xlix) 4500–S^{2-}, Sulfide. 2021. Table IB.

(l) 4500–SO_3^{2-}, Sulfite. 2021. Table IB.

(li) 4500–SO_4^{2-}, Sulfate. 2021. Table IB.

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(lv) 5520, Oil and Grease. 2021. Table IB.

(lvi) 5530, Phenols. 2021. Table IB.

(lvii) 5540, Surfactants. 2021. Table IB.

(lviii) 6200, Volatile Organic Compounds. 2020. Table IC.

(lix) 6410, Extractable Base/Neutrals and Acids. 2020. Tables IC and ID.

(lx) 6420, Phenols. 2020. Table IC.

(lxi) 6440, Polynuclear Aromatic Hydrocarbons. 2021. Table IC.

(lxii) 6630, Organochlorine Pesticides. 2021. Table IC.

(lxiii) 6640, Acidic Herbicide Compounds. 2021. Table IC.

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(lxvii) 9221, Multiple-Tube Fermentation Techniques for Members of the Coliform Group. 2014. Table IA, Notes 12, 14 and 33; Table IH, Notes 10, 12 and 32.

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(xi) ASTM D888–18, Standard Test Methods for Dissolved Oxygen in Water. May 2018. Table IB.

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(xx) ASTM D1293–18, Standard Test Methods for pH of Water. January 2018. Table IB.

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(xxx) ASTM D1976–20, Standard Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy. June 2020. Table IB.

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(xxxii) ASTM D2330–20, Standard Test Method for Methylene Blue Active Substances. February 2020. Table 1B.

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(lix) ASTM D5907–18, Standard Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water. May 2018. Table IB.

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(lxv) ASTM D7237–18, Standard Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection. January 2019. Table IB.

(lxvi) ASTM D7284–20, Standard Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection. August 2020. Table IB.

(lxvii) ASTM D7365–09a (Reapproved 2015), Standard Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide. August 2015. Table II, Notes 5 and 6.

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(lxix) ASTM D7573–18a^{e1}, Standard Test Method for Total Carbon and Organic Carbon in Water by High Temperature Catalytic Combustion and Infrared Detection, January 2019. Table IB.

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(33) Pace Analytical Services, LLC, 1800 Elm Street SE, Minneapolis, MN 55414. Telephone: 612–656–2240.

(i) PAM–16130–SSI, Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs) Using Shimadzu Gas Chromatography Mass Spectrometry (GC–MS/MS), Revision 1.1, May 20, 2022. Table IC, Note 17.

(ii) [Reserved]

(34) SGS AXYS Analytical Services, Ltd., 2045 Mills Road, Sidney, British Columbia, Canada, V8L 5X2. Telephone: 1–888–373–0881.

(i) ATM 16130, Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs) Using Waters and Agilent Gas Chromatography-Tandem-Mass Spectrometry (GC/MS/MS)., Revision 1.0, August 2020. Table IC, Note 16

(ii) [Reserved]

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

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[5] ASTM D7365–09a (15) 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.

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