PRE-PUBLICATION NOTICE

The EPA Administrator, Michael S. Regan, signed the following proposed rule on December 6, 2024, and EPA is submitting it for publication in the Federal Register (FR). It is not the official version of the proposed rule. This document is not disseminated for purposes of EPA's Information Quality Guidelines and does not represent an Agency determination or policy. While we have taken steps to ensure the accuracy of this internet version of this notice, the official version will be published in a forthcoming FR publication, which will appear on https://www.federalregister.gov and on Regulations.gov (https://www.regulations.gov) in Docket No. EPA-HQ-OW-2024-0328.

6560-50-P

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[EPA-HQ-OW-2024-0328; FRL 11799-01-OW]

RIN 2040-AG37

Clean Water Act Methods Update Rule 22 for the Analysis of Contaminants in Effluent

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule.

SUMMARY: The U.S. Environmental Protection Agency (EPA) is proposing to promulgate new methods and update the tables of approved methods for the Clean Water Act. The Clean Water Act requires the EPA to promulgate test procedures for the analysis of pollutants. Promulgating new methods and updating the tables of approved methods increases the quality and consistency of data collected for the purposes of the Clean Water Act. In this rule, the EPA proposes to add new EPA methods for per- and polyfluoroalkyl substances (PFAS) and polychlorinated biphenyl (PCB) congeners, and add methods previously published by voluntary consensus bodies that industries and municipalities would use for reporting under the EPA’s National Pollutant Discharge Elimination System permit program. The EPA also proposes to withdraw the seven Aroclor (PCB mixtures) parameters. In addition, the EPA is proposing to simplify the sampling requirements for two volatile organic compounds, and make a series of minor corrections to existing tables of approved methods. This proposed rule does not mandate when a parameter must be monitored or establish a discharge limit.

DATES: Comments must be received on or before **[INSERT DATE 30 DAYS AFTER DATE OF PUBLICATION IN THE FEDERAL REGISTER]**.

ADDRESSES: You may send comments, identified by Docket ID No. EPA-HQ-OW-2024-0328, by any of the following methods:

- Federal eRulemaking Portal: *https://www.regulations.gov/* (our preferred method). Follow the online instructions for submitting comments.
- 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: Adrian Hanley, Engineering and Analysis Division, Office of Water (4303T), Environmental Protection Agency, 1200 Pennsylvania Avenue NW, Washington, DC 20460-0001; telephone number: 202-564-1564; email address: *hanley.adrian@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–2024–0328, 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. The EPA may publish any comment received to its public docket. Do not submit to the 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. The EPA will generally not consider comments or comment contents located outside of the primary submission (i.e., on the web, cloud, or other file sharing system). Please visit *https://www.epa.gov/dockets* for additional submission information; 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 *https://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 action include:

Category	Examples of potentially affected entities
State, Territorial, and Indian Tribal Governments	States authorized to administer the National Pollutant Discharge Elimination System 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 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 the EPA is now aware of that could potentially be affected by this action. Other types of entities not listed in the table could also be affected. To determine whether your facility is affected by this action, you should carefully examine the applicability language at 40 CFR 122.1 (NPDES purpose and scope), 40 CFR 136.1 (NPDES permits and CWA) and 40 CFR 403.1 (pretreatment standards purpose and applicability). If you have questions regarding the applicability of this action to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

B. What Action is the Agency Taking?

Periodically, the EPA updates the approved methods in 40 CFR part 136. This proposed rulemaking is the 24th update to 40 CFR part 136 since its inception in 1973, 22 Method Update Rules and two Routine Method Update Rules. In general, the changes in this action fall into six categories. The first category is withdrawal of the seven Aroclor parameters and associated methods. The second category is withdrawal of outdated methods that have been supplanted by already approved methods at 40 CFR part 136. The third category is a simplification of the sampling and preservation requirements for two volatile organic compound methods. The fourth category includes minor corrections in the text and tables in 40 CFR part 136. The EPA is proposing these revisions to keep current with technology advances, and to improve data quality and consistency for the regulated community. The fifth category is new EPA methods for new parameters that are being added to 40 CFR part 136. The sixth category is new methods published by a voluntary consensus standard body or vendor for new parameters. The following paragraphs provide details on the revisions. The first four categories are discussed in section IV Corrections or Amendments to the Text and Tables of 40 CFR part 136 of this preamble, the fifth and sixth categories are discussed in section V Incorporation by Reference of this preamble.

C. What is the Agency's Authority for Taking this Action?

The EPA is proposing this regulation under the authorities of sections 301(a), 304(h), and 501(a) of the CWA; 33 U.S.C. 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, a NPDES permit issued under section 402 of the CWA. Section 304(h) of the CWA requires the 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 EPA Administrator to "… prescribe such regulations as are necessary to carry out this function under [the CWA]." The EPA generally has codified its test procedure regulations (including analysis and sampling requirements) for CWA programs at 40 CFR part 136, though some requirements are codified in other parts (e.g., 40 CFR Chapter I, Subchapters N and O).

III. Background

This preamble describes the abbreviations and acronyms used, the legal authority and reasons for the proposed rule, and a summary of the proposed changes and clarifications, and solicits comment from the public.

Abbreviations and Acronyms Used in the Preamble

AOF: Adsorbable Organic Fluorine

ASTM: ASTM International[1]

CFR: Code of Federal Regulations

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

CWA: Clean Water Act

EPA: The U.S. Environmental Protection Agency

GC/MS: Gas Chromatography/Mass Spectrometry

H_2O_2: Hydrogen Peroxide

ICP/AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy

LC-MS/MS: Liquid Chromatography-Tandem Mass Spectrometry (also used as LC/MS/MS in some methods)

NPDES: National Pollutant Discharge Elimination System

NTTAA: National Technology Transfer and Advancement Act

PAA: Peracetic Acid

PCB: Polychlorinated Biphenyl

PFAS: Per- and Polyfluoroalkyl Substances

POTW: Publicly Owned Treatment Works

QC: Quality Control

TNI: The NELAC Institute

USGS: United States Geological Survey

VCSB: Voluntary Consensus Standards Body

NPDES permits must include conditions 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. Permittees must use EPA-approved analytical methods that are sensitive enough to detect and measure pollutants at or below permit limits or water quality criteria (CWA section

402(b)(2)(A)). Permits include measurement and reporting requirements for these specific pollutants; however, permittees often have a choice of which approved test procedure they will use when the EPA has approved more than one method for a specific pollutant.

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, 40 CFR 403.12(b)(5)(v), and 40 CFR 403.12(g)(3).

Periodically, the EPA promulgates updates to the approved methods in 40 CFR part 136 to improve data quality and consistency. The changes proposed in this action fall into the following categories. The first category is withdrawal of seven Aroclor mixtures and the associated approved methods for Aroclors from the list of parameters at Table Ic that have been replaced with a method for the PCB congeners and total PCBs at Table Ii that provides a more accurate and complete characterization of the PCB content of samples than Aroclor analyses (see section V.A of this preamble). The second category is withdrawal of outdated methods that includes single-analyte total metal colorimetric methods for twelve metal parameters, a Waters^{TM} anion method, and EPA Method 1664A for the parameter "oil and grease" (see section IV.B of this preamble). The third category is simplifying sampling requirements for purgeable compounds by making the preservation requirement for acrolein and acrylonitrile consistent with the other pollutants analyzed by volatile organic compound methods (see section IV.C of this preamble). The fourth category is minor corrections to the text and tables at 40 CFR part 136 (see section IV.D of this preamble). The fifth category is new EPA methods for new parameters not previously listed at 40 CFR part 136 that will be added by incorporation by reference: per- and polyfluoroalkyl substances (PFAS) by EPA Method 1633A to a new Table Ij, adsorbable organic fluorine (AOF) by EPA Method 1621 to Table Ic, and polychlorinated biphenyl (PCB) congeners by EPA Method 1628 to a new Table Ii (see section V.A of this preamble). The EPA has finalized these three EPA methods and has posted these methods, along with their validation study reports, on EPA's CWA Methods website (*https://www.epa.gov/cwa-methods*). The sixth category is new methods published by the voluntary consensus standard bodies (VCSBs) or vendors for new parameters not previously listed at 40 CFR part 136 that will be added by incorporation by reference: PFAS analytes by ASTM D8421-24 to Table Ij; peracetic acid (PAA) by Standard Method "4500-PAA Peracetic Acid (Residual)," and hydrogen peroxide (H_2O_2) by Standard Method "4500-H_2O_2 Hydrogen Peroxide (Residual)" to Table Ib (see section V.B of this preamble). Finally, as a consequence of adding the two new tables for the PCB congeners and PFAS, the format of the numbers for the existing Tables IA through IH are being revised to be called out as Tables Ia through Ih. The use of a lowercase letter in the table identifier is necessary to avoid confusion between the new Table Ii of PCB congeners and the existing Table II with the required containers, preservation techniques, and holding times (see section V.A of this preamble).

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

A. Withdrawal of the Aroclor Parameters and the Associated Methods.

The EPA proposes to withdraw the seven Aroclor parameters listed as PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, and PCB-1260 and their associated methods from Table Ic, Parameters 88-94. Removing these parameters will result in the renumbering of all of the subsequent parameters in that table. This will also result in deletion of footnote 8, and the renumbering of footnotes 9 through 16. All of the methods listed for these parameters are listed elsewhere for other parameters, so none of them will be completely removed from 40 CFR part 136.3. The “PCBs” class of compounds is made up of 209 structurally related but distinct chemicals often called “congeners.” The seven Aroclor parameters are commercial PCB mixtures and were first included in the EPA’s 1976 Priority Pollutant List (Appendix A to Part 423, Title 40). These seven PCB mixtures were the only ones for which the EPA had valid methods at that time. However, none of these Aroclor mixtures include all the PCB congeners that may be present in an environmental sample. The EPA developed the original Aroclor methods in the 1970s, when analysis of individual PCB congeners was impractical with the existing affordable analytical technology and in the absence of automated laboratory information management systems. During the multi-laboratory study of Method 608 in the late 1970s, roughly 20% of the Aroclor data were rejected due to poor accuracy, and false negatives (non-detects) were an issue even with samples that were spiked with fresh standards.[2]

When used as a surrogate for PCBs, the results from the approved Aroclor methods can create false confidence that PCBs are not present. Given that the manufacturing and use of PCBs have been banned in the U.S. since 1979, most PCBs in the environment have been subjected to extensive changes in composition and concentration through processes that are collectively referred to as "weathering." Moreover, there are other relevant sources of PCBs to the environment beyond the seven Aroclor parameters that are not accounted for through Aroclor analyses. Aroclor methods were the best available technology when they were validated in 1978, but now there are much more accurate methods. Thus, the EPA is proposing to replace the Aroclor analysis with EPA Method 1628, which detects the 209 PCB congeners directly, either as individual congeners or groups of co-eluting congeners. As demonstrated by the EPA's multi-laboratory study[3], EPA Method 1628 can be widely implemented because it uses laboratory equipment that is readily available and is already employed by many full-service laboratories.

Withdrawal of the Aroclor parameters and associated methods will not have any immediate impact on current NPDES permits because permits that have been issued by the permitting authority can continue until they expire. 40 CFR 122.21(e)(3)(ii) states: "(ii) When there is no analytical method that has been approved under 40 CFR part 136,

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[2] EPA document EPA-600/4-84-061, "EPA Method Study 18 Method 608 – Organochlorine Pesticides and PCBs." June 1984.

[3] EPA document EPA 820-R-21-003, "Report on the Multi-laboratory Validation of Clean Water Act Method 1628 for PCB Congeners." April 2021.

required under 40 CFR chapter I, subchapter N or O, and is not otherwise required by the Director, the applicant may use any suitable method but shall provide a description of the method. When selecting a suitable method, other factors such as a method's precision, accuracy, or resolution, may be considered when assessing the performance of the method." EPA anticipates renewed permits will switch to the approved PCB congener method. Given that most permits are renewed every 5 years or less, promulgation of this part of this proposed rule would result in a multi-year phase out of Aroclor analysis.

B. Withdrawal of Outdated Methods

The EPA proposes to withdraw approval of outdated colorimetric methods for 12 individual metals and one inorganic anions alternate test procedure that the sponsoring organization no longer supports or makes available to the public. The removal of these methods and their associated footnotes will result in renumbering of the following footnotes at the end of Table Ib.

1. The EPA proposes to withdraw 12 single-metal colorimetric methodology types that measure a total metal concentration (e.g., "Aluminum—Total") from 40 CFR 136.3, Table Ib. There are multiple inductively coupled plasma (ICP) and graphite furnace methods that are more sensitive and provide more accurate results in challenging matrices that are already listed in Table Ib. These ICP and graphite furnace methods are also relatively inexpensive and easily automated, particularly when analyzing for one parameter (as is done for the colorimetric methods that the EPA is proposing to withdraw). The suggestion to withdraw these methods initially came from The NELAC Institute (TNI), which accredits a large portion of the environmental laboratories in the U.S. TNI is unaware of any laboratory that is currently accredited for these outdated

colorimetric methods. If the EPA receives compelling reason to not withdraw any of these methods, the EPA will consider keeping some or all of these methods in 40 CFR part 136. Otherwise, the EPA proposes to withdraw the methods in the rows where “Colorimetric” is the listed methodology for the following total metals, listed in order of the parameter number in Table Ib: 3. Aluminum—Total, 6. Arsenic—Total, 8. Beryllium—Total, 10. Boron—Total, 12. Cadmium—Total, 19. Chromium—Total, 22. Copper—Total, 30. Iron—Total, 32. Lead—Total, 34. Manganese—Total, 74. Vanadium—Total, and 75. Zinc—Total. In addition, footnotes pertaining to only these withdrawals will be removed and the remaining footnotes renumbered sequentially. The relevant methods that will be withdrawn are listed here by organization. Standard Methods: 3500-Al B-2020, 3500-As B-2020, 4500-B B-2011, 3500-Cd D-1990, 3500-Cr B-2020, 3500-Cu B and C-2020, 3500 Fe B-2011, 3500-Pb B-2020, 3500-Mn B-2020, 3500-V B-2011, 3500-Zn B-2020, and Footnote 61 that references an aluminon colorimetric method in Standard Methods 19th Edition. ASTM: D2972-15 (A), D1068-15 (C). USGS: Methods I-3060-85 and I-3112-85. Hach: Footnote 19 that references Method 8506, Bicinchoninate Method for Copper, Hach Handbook of Water Analysis, 1979; Footnote 22 that references Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, 1980; Footnote 23 that references Method 8034, Periodate Oxidation Method for Manganese, Hach Handbook of Wastewater Analysis, 1979; and Footnote 33 that references Method 8009, Zincon Method for Zinc, Hach Handbook of Water Analysis, 1979. AOAC International: Footnote 3 that references Method 920.203, the persulfate oxidation procedure from the Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth

Edition, 4th Revision, 1998.

2. The EPA also proposes to withdraw from Table Ib the Waters™ Corporation alternate test procedure: "Method D6508 Rev.2, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte." The Waters™ Corporation no longer supports this method and no longer provides copies of this method to the public. Waters™ developed their alternative test procedure to incorporate additional analysis techniques into an older ASTM standard. The method modifications developed by Waters™ were incorporated into later versions of the ASTM standard, including D6508-15, which is the currently approved method for the same anions: bromide, chloride, fluoride, nitrate, nitrate-nitrite, nitrite, orthophosphate, and sulfate (86 FR 27226, May 19, 2021). Both the Waters™ and ASTM methods are capillary ion electrophoresis methods that provide a simultaneous separation and determination of several inorganic anions and identifies all anions present in the sample by indirect UV detection. The EPA proposes to remove the eight occurrences of Method D6508 Rev. 2 from Table Ib. They are found in parameters: 11. Bromide, 16. Chloride, 25. Fluoride-Total, 38 Nitrate (as N), 39. Nitrate-nitrite (as N), 40. Nitrite (as N), 44. Ortho-phosphate (as P), and 65. Sulfate (as SO_4).

3. The EPA proposes to withdraw EPA Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGTHEM; Non-polar Material) by Extraction and Gravimetry, approved at 64 FR 26315, May 14, 1999, from Table Ib, parameter 41. A later revision of Method 1664, (1664 Rev. B), was approved by the EPA in a 2012 rulemaking, 77 FR 29758, May 18, 2012. That 2012 rule strongly recommended laboratories and permitting

authorities replace Method 1664, Rev. A with Method 1664, Rev. B. The 2012 rule also said that the EPA would revisit withdrawing Method 1664, Rev. A in a future rulemaking (77 FR 29758, May 18, 2012). Laboratories and regulatory entities have had more than 12 years to make this adjustment, so the EPA is proposing to withdraw Method, 1664, Rev. A from Table Ib, for parameter 41. Oil and Grease—Total recoverable.

C. Simplify the Sampling and Preservation Requirements for Two Purgeable Organic Compounds (Often Referred to as Volatile Organic Compounds)

The EPA proposes to change the sampling and preservation requirements for acrolein and acrylonitrile to match those of the other purgeable halocarbons and purgeable aromatic hydrocarbons in Table II under the section for parameters in Table Ic. The EPA proposes to change the preservation pH requirement from pH 4-5 to a pH of 2, and to use hydrochloric acid to adjust the sample pH, to align with the approved practice for other purgeable organic compounds. The Environmental Monitoring Coalition provided the EPA with the results from a study using multiple wastewater types that demonstrated that the analysis of acrolein and acrylonitrile is not adversely affected by the lower pH preservation. That Environmental Monitoring Coalition study report: “Evaluating Preservation Requirements for Acrolein and Acrylonitrile in Aqueous Matrices,” is provided in the docket. Thus, the EPA is proposing these changes to make the sampling, analysis, and data reporting more efficient for purgeable organic compound methods.

D. Minor Corrections and Amendments to the Texts and Tables of 40 CFR Part 136.

Stakeholders have identified several minor errors in Tables Ib, Id, and II. The EPA proposes to correct the following entries in those tables.

1. The EPA proposes to remove the row for the methodology "Amperometric direct (low level)" in Table Ib, parameter 17A, Chlorine-Free Available, because the only method listed in that row, Standard Method 4500 Cl E, measures Total Residual Chlorine (e.g., parameter 17), not free chlorine. The method will remain in the corresponding row for Residual Chlorine parameter 17, where the parameter matches the method.

2. The EPA proposes to amend Table Ib parameter 24 by adding 4500-CN$^-$P-2021 and D7511(17) to allow use of any method approved for measurement of Total Cyanide in both the untreated and treated fractions of a sample for determination of available cyanide by cyanide amenable to chlorination (CATC), provided the treatment steps in the two currently approved amenable cyanide methods, 4500-CN G-2016 and ASTM D2036-09(15)(B), are followed.

3. The EPA proposes to add EPA Method 625.1 as an approved method for the parameter Parathion ethyl, parameter 53 in Table Id. The use of EPA Method 625.1 was incorrectly omitted from Table Id for this parameter the last time that Table Id was updated (82 FR 40836, August 28, 2017) because the method lists this analyte as "Parathion," rather than Parathion ethyl. EPA Method 625.1 measures aqueous samples by extracting them with methylene chloride at pH 11 - 13 and again at a pH less than 2 using a separatory funnel or continuous liquid/liquid extractor. Then the extract is concentrated and analyzed by GC/MS.

4. The EPA proposes to apply footnote 9 to the entry for EPA Method 1613B in Table Ic for parameter 96, 2,3,7,8-Tetrachloro-dibenzo-*p*-dioxin (formerly listed a parameter 103). This footnote (formerly listed as footnote 10) was to be added to all 17 dioxin and furan congeners in a previous rule (88 FR 10739, February 21, 2023) but was

inadvertently missed for this parameter.

5. The EPA proposes to add sodium thiosulfate, $Na_2S_2O_3$, to the preservation column for parameter 4, Ammonia, under Table Ib in Table II, and apply Footnote 5 to that entry to address the fact that residual chlorine in samples will react with ammonia to form chloramines, which are an interference with ammonia analyses. The EPA also is adding the following text to the end of Footnote 5, “Section A.2 of Standard Method 4500-NH3 discusses the need for dechlorination of samples for ammonia analysis.”

6. The EPA proposes to edit Footnote 17 to Table II under the section for parameters in Table Ib, as applied to parameter 35 for “Mercury (CVAFS)” to include mention of EPA Method 245.7 along with EPA Method 1631 in the footnote. Both methods employ the same cold vapor atomic fluorescence spectroscopy (CVAFS) determinative technique and including EPA Method 245.7 in the footnote will help clarify that the 90-day holding time applies to EPA Method 245.7 as well as EPA Method 1631. Currently, Method 245.7 is not specifically mentioned in Table II, which was an oversight.

7. The EPA proposes to specify in Table II that sodium thiosulfate is to be added to samples to remove oxidants (e.g., chlorine) for the Table 1B parameter 48 (Phenols) and the Table 1C parameters 107 to 111 (Alkylated phenols). The EPA also proposes to restore footnote 5 in Table II for Table 1C parameters 23, 30, 44, 49, 53, 77, 80, 81, 91, 93, and 105 (Phenols), and to add Footnote 5 for Table 1C parameters 112 (Adsorbable Organic Halides) and 113 (Chlorinated Phenolics), and to clarify that the addition of sodium thiosulfate is only necessary when oxidants are present (e.g., chlorine).

8. The EPA proposes to add a new footnote to Table II under Table Ib and apply

it to the entry for parameter 50, Phosphorus—Total. The new Footnote 25 addresses the fact that many laboratories determine total phosphorus using EPA Method 200.7, an ICP/AES procedure for metals that is also approved for phosphorus. However, as laboratories often note, the acid specified in Table II to preserve metals samples is nitric acid, where Table II calls out sulfuric acid for phosphorous. In both cases the specified pH is a value less than or equal to 2. In addition, while Table II allows acidified samples to held for 6 months, it only allows phosphorus samples to be held for 28 days. The new Footnote 25 reads: “When EPA Method 200.7 or another approved ICP/AES method is used to determine total phosphorus, sulfuric acid may be used to preserve the sample to pH ≤ 2, or the acid-preserved sample for metals may be used for analysis, and the holding time for total phosphorus may be extended to 6 months.”

V. Incorporation by Reference

Currently, hundreds of methods and alternate test procedures 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 VCSB methods that are incorporated by reference (see section V.B of this preamble). The proposed VCSB methods are consistent with the requirements of the National Technology Transfer and Advancement Act (NTTAA), under which Federal agencies should use technical standards developed or adopted by the VCSBs if compliance would not be inconsistent with applicable law or otherwise impracticable (see section VI 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 includes three new EPA methods (see section V.A of this preamble) and several vendor and VCSB methods for four parameter types (see section V.B of this preamble) which the EPA proposes to incorporate by reference. The EPA methods and the vendor methods are available free of charge on their respective websites, therefore the EPA methods and vendor methods incorporated by reference are reasonably available.

A. Changes to 40 CFR 136.3 to Include Three New EPA Methods and their Associated Parameters

The EPA proposes to add three new EPA methods and their associated parameters to 40 CFR 136.3: EPA Method 1621: Determination of Adsorbable Organic Fluorine (AOF) in Aqueous Matrices by Combustion Ion Chromatography (CIC), EPA Method 1628: Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-resolution GC/MS using Selected Ion Monitoring, and EPA Method 1633A: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. All three of these methods have multi-laboratory validation studies that demonstrate they perform well in a wide variety of wastewater types at multiple laboratories. These methods include performance (or quality control) criteria that the EPA generated by statistically analyzing the data from the multi-laboratory validation studies. The multi-laboratory validation study reports and methods are available to the public on the EPA's Clean Water Act Analytical Methods website at https://www.epa.gov/cwa-methods, and in the docket to this proposed rulemaking.

Adding these methods and parameters to 40 CFR part 136 does not require the permitting authority to require monitoring for those parameters, but it informs the permitting authority and discharger that there is an available method that has been validated and tested. Once these methods are finalized in 40 CFR part 136, then use of the approved test procedure is required if any of these parameters are added to a NPDES permit. The EPA proposes to add the parameter "Adsorbable Organic Fluorine," (AOF) to Table Ic with the approved test procedure EPA Method 1621. EPA Method 1621 determines adsorbable organic fluorine by passing an aqueous sample through two granular activated carbon columns. The columns are rinsed with sodium nitrate to remove inorganic fluoride, combusted at ≥1000 °C in an oxygen or oxygen/argon stream, and the gaseous hydrogen fluoride is absorbed into reagent water. The fluoride is separated by ion chromatography (IC), identified by comparing sample fluoride retention time to retention times for calibration standards acquired under identical Ic conditions, and then quantified using the external standard technique.

The EPA proposes to redesignate all tables in 136.3 with a lower case letter, for example Table IA would be "Table Ia—List of Approved Biological Methods for Wastewater and Sewage Sludge." This redesignation of the table titles is needed to avoid confusion with the currently existing Table II (Roman numeral two) and the new table for PCBs in order to distinguish between Roman numeral one followed by the letter "I" and Roman numeral two. Then Tables Ii and Ij will be added, as discussed below. The number format for Table II—Required Containers, Preservation Techniques, and Holding

Times will remain unchanged.

The EPA proposes to add the 209 Polychlorinated Biphenyl (PCB) Congeners and Total PCBs as new parameters in a new Table Ii with the approved test procedure EPA Method 1628. Method 1628 detects all PCB 209 congeners, which are all the compounds that make up the class of chemicals called “PCBs.” The new parameter called “Total PCBs” will be the summation of all the detected PCB congeners in the sample as determined by Method 1628. The summation will use zero (0) for the contribution of the congeners that are not detected. Otherwise, every method blank would have a non-zero result, even when no congeners are detected, affecting assessments of the sample results relative to the method blank. There is no method detection limit (MDL) for Total PCBs because it is the sum of the results for many individual parameters. Method blanks and samples with no detects are reported as “ND” for non-detect, or a similarly appropriate non-numeric value. Method 1628 analyzes samples by spiking them with isotope dilution standards, extracting the PCBs using separatory funnel extraction or solid-phase extraction, cleaning the extracts with a Florisil® column, and analyzing the sample extracts using GC/MS with selected ion monitoring. Because the full names of the PCB congeners are long and contain many numbers indicating the positions of the chlorine substituents (e.g., 2,3,3',4,4',5-Hexachlorobiphenyl), they are often expressed as a “congener number” in the format “PCB-XYZ,” where “XYZ is a number between 1 and 209. The EPA has included the congener numbers in Table Ii to facilitate consistency in reporting of the results for these new parameters.

The EPA proposes to add the 40 target PFAS analytes in Method 1633A as new parameters in a new Table Ij, with the approved test procedure EPA Method 1633A.

Method 1633A analyzes samples by spiking them with isotope dilution standards, subjecting them to solid-phase extraction and activated carbon cleanup, and analyzing the sample extracts using liquid chromatography-tandem mass spectrometry (LC-MS/MS).

The EPA proposes to add these new parameters to the relevant section of Table II—Required Containers, Preservation Techniques, and Holding Times. The specifications for the containers, preservation techniques, and holding times for these parameters are the same as the specifications provided in the method.

B. Changes to 40 CFR 136.3 to Include New Parameters and Methods Published by VCSBs and Vendors

The EPA solicited VCSBs and relevant vendors to submit methods, along with study reports and validation data, for the following parameters: the PFAS parameters listed in Method 1633A, peracetic acid, and hydrogen peroxide. The VCSBs or vendors submitted data that demonstrate that their methods can be used at multiple laboratories and can reliably detect and quantify the parameter in a wide variety of wastewater types (as the EPA does with its own CWA methods). The methods and multi-laboratory study reports for each of the following methods are in the docket for this rulemaking. The EPA proposes to add the following parameters and methods to 40 CFR part 136.

The EPA also proposes to add these new parameters to the relevant section of Table II—Required Containers, Preservation Techniques, and Holding Times. The specifications for the containers, preservation techniques, and holding times for peracetic acid, and hydrogen peroxide are the same as the specifications provided in the methods. The preservation and holding time specifications for the PFAS parameters parallel those from EPA Method 1633A.

1. "ASTM D8421-24 Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices by Co-solvation followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)," as an approved test procedure for the 40 PFAS analytes being added as new parameters in Table Ij. ASTM D8421-24 analyzes aqueous samples by adding labeled surrogates to a 5-mL sample in the original container, adding 5 mL of methanol, filtering the sample, adjusting the pH and injecting a small aliquot of the extract into an LC-MS/MS instrument.

2. The EPA proposes to add the parameters "Peracetic acid (PAA)," and "Hydrogen Peroxide (H_2O_2)," to Table Ib and approve Standard Method "4500-PAA Peracetic Acid (Residual)," and Standard Method "4500-H_2O_2 Hydrogen Peroxide (Residual)." Both methods use simple handheld instruments that use a chemical indicator followed by colorimetric detection. They are similar to the colorimetric methods used for residual chlorine. These two new parameters will provide POTWs and other wastewater treatment permittees that utilize peracetic acid as an alternative to chlorine-based disinfectants with approved methods to monitor their discharges.

VI. Statutory and Executive Order Reviews

A. Executive Order 12866: Regulatory Planning and Review Executive Order 14094: Modernizing Regulatory Review

This action is not a significant regulatory action as defined in Executive Order 12866, as amended by Executive Order 14094, and was therefore not subject to a requirement for Executive Order 12866 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

I certify that this action would not have a significant economic impact on a substantial number of small entities under the Regulatory Flexibility Act. This action would not impose any requirements on small entities. This action would approve new and revised versions of CWA testing procedures. Generally, these changes improve data quality. In general, the EPA expects the proposed revisions would lead to few direct costs. Adding these parameters and methods to 40 CFR part 136 does not require the permitting authority to require monitoring that parameter, but it informs the permitting authority and discharger that there is an available method that has been validated and tested in a wide variety of wastewater types. If a permittee is already monitoring for one of more of the parameters, then the permittee may need to change methods. This would not be a significant change in cost, given that the permittee is already paying a laboratory to analyze for the parameter(s). The EPA proposes methods that would be incorporated by reference. If a permittee elected to use these methods, they could incur a small cost associated with obtaining these methods from the listed sources. See section IV of this preamble.

D. Unfunded Mandates Reform Act

This action does not contain an 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. The EPA does not expect the proposal would lead to any costs to any tribal governments, and if incurred, the 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 the EPA has reason to believe may disproportionately affect children, per the definition of “covered regulatory action” in section 2-202 of the Executive Order. Therefore, this action is not subject to Executive Order 13045 because it does not concern an environmental health risk or safety risk. Since this action does not concern human health, the EPA's policy on Children's Health also does not apply.

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

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

I. National Technology Transfer and Advancement Act of 1995

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

J. Executive Order 12898: Federal Actions to Address Environmental Justice in Minority Populations and Low-Income Populations and Executive Order 14096: Revitalizing Our Nation's Commitment to Environmental Justice for All

The 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 communities with environmental justice concerns.

This action has no effect on communities because this action will approve new CWA testing procedures. These changes would provide increased flexibility for the regulated community in meeting monitoring requirements while improving data quality. Adding new parameters to 40 CFR part 136 does not require the parameters to be monitored. In addition, this update to the CWA methods will incorporate technological advances in analytical technology. Although this action does not concern human health or environmental conditions, the EPA identifies and addresses environmental justice concerns 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.

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 forth 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 by:

a. In paragraph (a):

i. Revising the introductory text;

ii. Revising the newly designated tables “Ib” and “Ic”;

iii. Revising the newly designated table “Id” by removing in the table heading, third column, the text “EPA [2 7 10]” and adding in its place the text “EPA [2, 7, 10]”; revising entry “53”;

iv. Add Tables “Ii” and “Ij”; and

b. In paragraph (b):

i. Revising the introductory text;

ii. Redesignating paragraphs (b)(8)(xi) through (xvi) as paragraphs (b)(8)(xiv) through (xix) and adding new paragraphs (b)(8)(xi) through (xiii);

iii. Revising and republishing paragraph (b)(10);

iv. Removing paragraph (b)(13)(i)(A), and redesignate paragraphs (b)(13)(B) through (X) as paragraphs (b)(13)(A) through (W);

v. Adding paragraph (b)(15)(lxxi);

vi. Removing paragraphs (b)(20)(ii) through (v), and redesignate paragraphs (b)(20)(vi) through (x) as paragraphs (b)(ii) through (vi);

vii. Revising paragraph (b)(38)(i); and

viii. Removing paragraph (b)(41).

c. In paragraph (c), amend Table II:

i. Under Table Ib-Inorganic Tests by revising entries “4”, “48” and “50”; and adding entries “77” and “78”;

ii. Revising the table entitled “Table Ic-Organic Tests”;

iii. Adding two tables immediately after the entry “7. *Giardia”* entitled “Table Ii—Polychlorinated Biphenyl Congener Tests” and “Table Ij—Per- and Polyfluorinated Alkyl Substances Tests”;

vi. Revising footnote “5”; and

v. Adding footnotes “25” through “29”.

The revisions and additions read as follows:

§ 136.3 Identification of test procedures

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables Ia, Ib, Ic, Id, Ie, If, Ig, Ih, Ii, and Ij of this section. The methods listed in Tables Ia, Ib, Ic, Id, Ie, If, Ig, Ih, Ii, and Ij are incorporated by reference, see paragraph (b) of this section, with the exception of EPA Methods 200.7, 601-613, 624.1, 625.1, 1613, 1624, and 1625. The full texts of Methods 601-613, 624.1, 625.1, 1613, 1624, and 1625 are printed in appendix A of this part, and the full text of Method 200.7 is printed in appendix C of this part. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part. In the event of a conflict between the reporting requirements of 40 CFR parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of 40 CFR parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full texts of the referenced test procedures are incorporated by reference into Tables Ia, Ib, Ic, Id, Ie, If, Ig, Ih, Ii, and Ij. The year after the method number indicates the latest editorial change of the method. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables Ia, Ib, Ic, Id, Ie, If, Ig, Ih, Ii, and Ij, or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under certain circumstances (paragraph (c) of this section, in § 136.5(a) through (d) or 40 CFR 401.13) other additional or alternate test procedures may be used.

*   *   *   *   *

TABLE Ib—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	**Methodology** [53]	**EPA** [48]	**Standard Methods** [78]	**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 [32]		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 [32]	200.5, Rev 4.2 (2003) [62], 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	Direct Current Plasma (DCP) [32]			D4190-15	See footnote [30]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
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 [55]
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction-based methods	350.1 [27], Rev. 2.0 (1993)	4500-NH_3 G-2021 4500-NH_3 H-2021		I-4523-85 [2], I-2522-90 [74]
	Automated electrode				See footnote.[7]
	Ion Chromatography			D6919-17	
	Automated gas diffusion, followed by conductivity cell analysis				Timberline Ammonia-001 [68]
	Automated gas diffusion followed by fluorescence detector analysis				FIAlab100 [76]
5. Antimony—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		3111 B-2019		
	AA furnace		3113 B-2020		
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62]; 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-4472-97 [75]
6. Arsenic—Total,[4] mg/L	Digestion [4], followed by any of the following:	206.5 (Issued 1978) [1]			

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	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 [45]
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62]; 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 [64]
7. Barium—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		3111 D-2019		I-3084-85 [2]
	AA furnace		3113 B-2020	D4382-18	
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020		I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP [32]				See footnote [30]
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) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP			D4190-15	See footnote [30]
9. Biochemical oxygen demand (BOD_5), mg/L	Dissolved Oxygen Depletion		5210 B-2016 [79]		973.44 [3], p. 17 [9], I-1578-78 [8], See footnotes [10, 57]
10. Boron—Total,[33] mg/L	ICP/AES	200.5, Rev 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]
	DCP			D4190-15	See footnote [30]
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 [73]
	CIE/UV		4140 B-2020	D6508-15	
12. Cadmium—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		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 [47]
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-1472-85 [2] or I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
	Voltammetry [11]			D3557-17 (C)	

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
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) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020		I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]
	DCP				See footnote [30]
	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 [79]		See footnotes .[31, 57]
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, 77], 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 [47]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	CIE/UV		4140 B-2020	D6508-15	
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	
	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]
19. Chromium—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		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 [42]
	STGFAA	200.9, Rev. 2.2 (1994)			

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62], 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 [64] I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
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 [47]
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4020-05 [64] I-4472-97 [75]
	DCP			D4190-15	See footnote [30]
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity	Colorimetric (ADMI)		2120 F-2021 [72]		
	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 [32]		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 [47]
	STGFAA	200.9, Rev. 2.2 (1994)			

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4020-05 [64], I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
23. Cyanide—Total, mg/L	Automated UV digestion/distillation and Colorimetry				Kelada-01. [50]
	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) [52]	4500-CN^- B-2021 and C-2021	D2036-09(15)(A), D7284-20	10-204-00-1-X [51]
	Flow Injection, gas diffusion amperometry			D2036-09(15)(A) D7284-20	
	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 [19]	335.4, Rev. 1.0 (1993) [52]	4500-CN^- N-2021		10-204-00-1-X [51], 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)	
	Cyanide Amenable to Chlorination (CATC) by difference; Segmented Flow Injection, In-Line Ultraviolet Digestion, followed by gas diffusion amperometry[81]		4500-CN^- P-2021	D7511-12(17)	

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	Flow injection and ligand exchange, followed by gas diffusion amperometry [54]		4500-CN$^-$ Q-2021	D6888-16	OIA-1677-09 [40]
	Automated Distillation and Colorimetry (no UV digestion)				Kelada-01 [50]
24.A Cyanide—Free, mg/L	Flow Injection, followed by gas diffusion amperometry		4500-CN$^-$ R-2021	D7237-18 (A)	OIA-1677-09 [40]
	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	
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 [30]
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]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	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 [20], I-2587-85 [2]
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 [32]		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 [32]	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]
	DCP [32]			D4190-15	See footnote [30]
31. Kjeldahl Nitrogen [5] - Total, (as N), mg/L	Manual digestion [19] 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 [41]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	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 [55]
	Automated gas diffusion, followed by conductivity cell analysis				Timberline Ammonia-001 [68]
	Automated gas diffusion followed by fluorescence detector analysis				FIAlab 100 [76]
	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 [41]
	Block digester, followed by Auto distillation and Titration				See footnote [35]
	Block digester, followed by Auto distillation and Nesslerization				See footnote [36]
	Block Digester, followed by Flow injection gas diffusion (distillation not required)				See footnote [37]
	Digestion with peroxydisulfate, followed by Spectrophotometric (2,6-dimethylphenol)				Hach 10242 [70]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	Digestion with persulfate, followed by Colorimetric				NCASI TNTP W10900 [71]
32. Lead—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		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 [47]
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES [32]	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
	Voltammetry [11]			D3559-15 (C)	
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) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]
	DCP				See footnote [30]
	Ion Chromatography			D6919-17	
34. Manganese—Total,[4] mg/L	Digestion[4], followed by any of the following:				
	AA direct aspiration [32]		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)	

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES [32]	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
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 [65]
	Purge and Trap CVAFS	1631E [39]			
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 [43]
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP				See footnote [30]
37. Nickel—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		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 [47]
	STGFAA	200.9, Rev. 2.2 (1994)			

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	ICP/AES [32]	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4020-05 [64], I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
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	
	Ion Selective Electrode		4500-NO_3^- D-2019		
	Colorimetric (Brucine sulfate)	352.1 (Issued 1971)[1]			973.50 [3], 419D [80], p. 28 [9]
	Spectrophotometric (2,6-dimethylphenol)				Hach 10206 [69]
	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 [47]
	Automated hydrazine		4500-NO_3^- H-2019		
	Reduction/Colorimetric				See footnote [56]
	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	

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	Enzymatic reduction, followed by automated colorimetric determination			D7781-14	I-2547-11 [66] I-2548-11 [66] N07-0003 [67]
	Enzymatic reduction, followed by manual colorimetric determination		4500-NO$_3^-$ J-2018		
	Spectrophotometric (2,6-dimethylphenol)				Hach 10206 [69]
40. Nitrite (as N), mg/L	Spectrophotometric: Manual		4500-NO$_2^-$ B-2021		See footnote [22]
	Automated (Diazotization)				I-4540-85 [2], See footnote [56] I-2540-90 [74]
	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	
	Automated (*bypass Enzymatic reduction)			D7781-14	I-2547-11 [66] I-2548-11 [66] N07-0003 [67]
41. Oil and grease—Total recoverable, mg/L	Hexane extractable material (HEM): *n*-Hexane extraction and gravimetry	1664 Rev. B [38]	5520 B or G-2021 [34]		
	Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry	1664 Rev. B [38]	5520 B or G-2021 [34] and 5520 F-2021 [34]		
42. Organic carbon—Total (TOC), mg/L	Combustion		5310 B-2014	D7573-18a^{e1}	973.47 [3], p. 14 [21]
	Heated persulfate or UV persulfate oxidation		5310 C-2014 5310 D-2011	D4839-03(17)	973.47[3],, p. 14 [21]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
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 [74]
	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	
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 footnotes [57], [58]
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 [30]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
48. Phenols, mg/L	Manual distillation [23], 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 [24]	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 [25]
50. Phosphorus—Total, mg/L	Digestion [19], 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, 32]	200.7, Rev. 4.4 (1994)	3120 B-2020		I-4471-97 [46]
	Semi-automated block digestor (TKP digestion)	365.4 (Issued 1974) [1]		D515-88 (B)	I-4610-91 [44]
	Digestion with persulfate, followed by Colorimetric				NCASI TNTP W10900 [71]
51. Platinum—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration		3111 B-2019		
	AA furnace	255.2 (Issued 1978) [1]			
	ICP/MS		3125 B-2020		
	DCP				See footnote [30]
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		

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	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 [45]
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES [32]	200.5, Rev 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4020-05 [64] I-4472-97 [75]
	AA gaseous hydride		3114 B-2020, or 3114 C-2020	D3859-15 (A)	I-3667-85 [2]
61. Silica—Dissolved, [33] 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) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020		I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]
62. Silver—Total,[4, 28] mg/L	Digestion [4, 26], 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 [47]
	STGFAA	200.9, Rev. 2.2 (1994)			
	ICP/AES	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4472-97 [75]
	DCP				See footnote [30]
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]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	ICP/AES	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020		I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]
	DCP				See footnote [30]
	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 [64]
	CIE/UV		4140 B-2020	D6508-15	
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	

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
69. Temperature, ° C	Thermometric		2550 B-2010		See footnote [29]
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 [46] I-4472-97 [75]
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) [62]; 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)			
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3]

Parameter	Methodology [53]	EPA [48]	Standard Methods [78]	ASTM	USGS/AOAC/ Other
	DCP				See footnote [30]
73. Turbidity, NTU [49]	Nephelometric	180.1, Rev. 2.0 (1993)	2130 B-2020	D1889-00	I-3860-85 [2] See footnotes [59, 60, 61]
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) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4020-05 [64]
	DCP			D4190-15	See footnote [30]
75. Zinc—Total,[4] mg/L	Digestion [4], followed by any of the following:				
	AA direct aspiration [32]		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 [32]	200.5, Rev. 4.2 (2003) [62]; 200.7, Rev. 4.4 (1994)	3120 B-2020	D1976-20	I-4471-97 [46]
	ICP/MS	200.8, Rev. 5.4 (1994)	3125 B-2020	D5673-16	993.14 [3], I-4020-05 [64], I-4472-97 [75]
	DCP [32]			D4190-15	See footnote [30]
76. Acid Mine Drainage		1627 [63]			
77. Hydrogen Peroxide, mg/L	Spectrophotometric, Ferric Thiocyanate		4500-H_2O_2 B-2020		
78. Peracetic Acid, mg/L	Spectrophotometric, DPD		4500-PAA B-2019		

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

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

[21] 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.

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

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

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

[25] Addison, R.F., and R.G. Ackman. 1970. Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography, *Journal of Chromatography*, 47(3):421-426.

[26] 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.

[27] 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.

[28] 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.

[29] "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.

[30] 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.

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

[32] 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.

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

[34] 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.

[35] Method PAI-DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric

Detection. Revised December 22, 1994. OI Analytical.

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

[37] Method PAI-DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion. Revised December 22, 1994. OI Analytical.

[38] Method 1664 Rev. B, *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, February 2010, U.S. EPA.

[39] 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.

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

[41] 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.

[42] 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.

[43] 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.

[44] 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.

[45] 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.

[46] 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.

[47] 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.

[48] 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.

[49] 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.

[50] 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.

[51] 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.

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

[53] 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.

[54] Samples analyzed for available cyanide using OI Analytical method OIA-1677-09 or ASTM method D6888-16 that contain particulate matter may be filtered prior to the ligand exchange reagents have been added to the samples.

[55] 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.

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

[57] 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).

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

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

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

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

[62] 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.

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

[64] 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.

[65] 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.

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

[67] NECi Method N07-0003, “Nitrate Reductase Nitrate-Nitrogen Analysis,” Revision 9.0, March 2014, The Nitrate Elimination Co., Inc.

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

[69] Hach Company Method 10206, “Spectrophotometric Measurement of Nitrate in Water and Wastewater,” Revision 2.1, January 2013, Hach Company.

[70] Hach Company Method 10242, “Simplified Spectrophotometric Measurement of Total Kjeldahl Nitrogen in Water and Wastewater,” Revision 1.1, January 2013, Hach Company.

[71] 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.

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

[73] 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.

[74] 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.

[75] 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.

[76] 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.

[77] 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.

[78] 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.

[79] 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.

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

[81] Prior to in-line UV digestion followed by gas diffusion amperometry, the sample must be treated as specified in SM 4500-CN- P-2021 (4a, b, and c) or ASTM D2036-09(15) (B, steps 21.1.1 through 21.1.5)

TABLE Ic—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE Organic COMPOUNDS

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
1. Acenaphthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
2. Acenaphthylene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] 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 [12]
5. Anthracene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
6. Benzene	GC	602	6200 C-2020		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
7. Benzidine	Spectrophotometric				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 [8] 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 [8] 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 [8] p. 27

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	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 [8] 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 [8] 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 [8] p. 27

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
15. bis(2-Chloroethoxy) methane	GC	611			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
16. bis(2-Chloroethyl) ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
17. bis(2-Ethylhexyl) phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
18. Bromodichloromethane	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
19. Bromoform	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
20. Bromomethane	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
21. 4-Bromophenyl phenyl ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] 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 [12], O-4436-16 [13]
23. 4-Chloro-3-methyl phenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] 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 [12] O-4436-16 [13]
25. Chloroethane	GC	601	6200 C-2020		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12]
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 [12], O-4436-16 [13]
28. Chloromethane	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
29. 2-Chloronaphthalene	GC	612			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
30. 2-Chlorophenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
31. 4-Chlorophenyl phenyl ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
32. Chrysene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] 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 [8] 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 [12], O-4436-16 [13]
35. 1,2-Dichlorobenzene	GC	601, 602	6200 C-2020		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	624.1, 1625B	6200 B-2020		See footnote [8] p. 27, O-4127-96 [12], O-4436-16 [13]
36. 1,3-Dichlorobenzene	GC	601, 602	6200 C-2020		
	GC/MS	624.1, 1625B	6200 B-2020		See footnote [8] p. 27, O-4127-96 [12]
37. 1,4-Dichlorobenzene	GC	601, 602	6200 C-2020		
	GC/MS	624.1, 1625B	6200 B-2020		See footnote [8] p. 27, O-4127-96 [12], O-4436-16 [13]
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 [12], O-4436-16 [13]
40. 1,1-Dichloroethane	GC	601	6200 C-2020		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
41. 1,2-Dichloroethane	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
42. 1,1-Dichloroethene	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
43. *trans*-1,2-Dichloroethene	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
44. 2,4-Dichlorophenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
45. 1,2-Dichloropropane	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12] O-4436-16 [13]

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
46. *cis*-1,3-Dichloropropene	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
47. *trans*-1,3-Dichloropropene	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
48. Diethyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
49. 2,4-Dimethylphenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
50. Dimethyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
51. Di-*n*-butyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27

Parameter [1]	Method	EPA [2, 7]	Standard Methods [16]	ASTM	Other
52. Di-*n*-octyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
53. 2, 4-Dinitrophenol	GC	604	6420 B-2021		See footnote [8] 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 [8] p. 27
55. 2,6-Dinitrotoluene	GC	609			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] 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 [12], O-4436-16 [13]
58. Fluoranthene	GC	610			

Parameter [1]	**Method**	**EPA [2, 7]**	**Standard Methods [16]**	**ASTM**	**Other**
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
59. Fluorene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
62. 1,2,3,4,6,7,8-Heptachloro-dibenzo-*p*-dioxin	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
63. Hexachlorobenzene	GC	612			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27

Parameter [1]	Method	EPA [2, 7]	Standard Methods [16]	ASTM	Other
64. Hexachlorobutadiene	GC	612			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27, O-4127-96 [12]
65. Hexachlorocyclopentadiene	GC	612			
	GC/MS	625.1 [5], 1625B	6410 B-2020		See footnote [8], p. 27, O-4127-96 [12]
66. 1,2,3,4,7,8-Hexachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
67. 1,2,3,6,7,8-Hexachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
68. 1,2,3,7,8,9-Hexachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
69. 2,3,4,6,7,8-Hexachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
70. 1,2,3,4,7,8-Hexachloro-dibenzo-*p*-dioxin	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
71. 1,2,3,6,7,8-Hexachloro-dibenzo-*p*-dioxin	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
72. 1,2,3,7,8,9-Hexachloro-dibenzo-*p*-dioxin	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
73. Hexachloroethane	GC	612			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27, O-4127-96 [12]
74. Indeno(1,2,3-c,d) pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
75. Isophorone	GC	609			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] 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 [12], O-4436-16 [13]

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
77. 2-Methyl-4,6-dinitrophenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
78. Naphthalene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021		
79. Nitrobenzene	GC	609			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC			D4657-92 (98)	
80. 2-Nitrophenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
81. 4-Nitrophenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
82. N-Nitrosodimethylamine	GC	607			
	GC/MS	625.1 [5], 1625B	6410 B-2020		See footnote [8] p. 27
83. N-Nitrosodi-*n*-propylamine	GC	607			
	GC/MS	625.1 [5], 1625B	6410 B-2020		See footnote [8] p. 27
84. N-Nitrosodiphenylamine	GC	607			
	GC/MS	625.1 [5], 1625B	6410 B-2020		See footnote [8] p. 27
85. Octachlorodibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
86. Octachlorodibenzo-*p*-dioxin	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
87. 2,2´-oxybis(1-chloropropane)[11] [also known as bis(2-Chloro-1-methylethyl) ether]	GC	611			

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
88. 1,2,3,7,8-Pentachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
89. 2,3,4,7,8-Pentachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
90. 1,2,3,7,8-Pentachloro-dibenzo-*p*-dioxin	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
91. Pentachlorophenol	GC	604	6420 B-2021		See footnote [3] p. 140
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
92. Phenanthrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
93. Phenol	GC	604	6420 B-2021		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
94. Pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
	HPLC	610	6440 B-2021	D4657-92 (98)	
95. 2,3,7,8-Tetrachloro-dibenzofuran	GC/MS	1613B [9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
96. 2,3,7,8-Tetrachloro-dibenzo-*p*-dioxin	GC/MS	613, 625.1 [5], 1613B[9]			SGS AXYS 16130 [14], PAM 16130-SSI [15]
97. 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 [12]
98. Tetrachloroethene	GC	601	6200 C-2020		See footnote [3] p. 130
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
99. Toluene	GC	602	6200 C-2020		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
100. 1,2,4-Trichlorobenzene	GC	612			See footnote [3] p. 130
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27, O-4127-96 [12], O-4436-16 [13]
101. 1,1,1-Trichloroethane	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
102. 1,1,2-Trichloroethane	GC	601	6200 C-2020		See footnote [3] p. 130
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
103. Trichloroethene	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
104. Trichlorofluoromethane	GC	601	6200 C-2020		

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
	GC/MS	624.1	6200 B-2020		O-4127-96 [12]
105. 2,4,6-Trichlorophenol	GC	604	6420 B-2021		
	GC/MS	625.1, 1625B	6410 B-2020		See footnote [8] p. 27
106. Vinyl chloride	GC	601	6200 C-2020		
	GC/MS	624.1, 1624B	6200 B-2020		O-4127-96 [12], O-4436-16 [13]
107. Nonylphenol	GC/MS			D7065-17	
108. Bisphenol A (BPA)	GC/MS			D7065-17	
109. *p-tert*-Octylphenol (OP)	GC/MS			D7065-17	
110. Nonylphenol Monoethoxylate (NP1EO)	GC/MS			D7065-17	
111. Nonylphenol Diethoxylate (NP2EO)	GC/MS			D7065-17	

Parameter [1]	**Method**	**EPA** [2, 7]	**Standard Methods** [16]	**ASTM**	**Other**
112. Adsorbable Organic Halides (AOX)	Adsorption and Coulometric Titration	1650 [10]			
113. Chlorinated Phenolics	In Situ Acetylation and GC/MS	1653 [10]			
114. Adsorbable Organic Fluorine (AOF)	Adsorption and Combustion Ion Chromato-graphy (CIC)	1621[17]			

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.

[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. Method 625.1 may be applied to 2,3,7,8-Tetrachloro-dibenzo-p-dioxin for screening purposes 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] 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.

[9] 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.

[10] 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.

[11] 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.

[12] 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.

[13] 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.

[14] SGS AXYS Method 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.

[15] 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: pacelabs.com.

[16] Please refer to the following applicable Quality Control Section: Part 6000 Individual Organic

Compounds, 6020 (2019). The Quality Control Standards are available for download at standardmethods.org at no charge.

[17] Method 1621, Determination of Adsorbable Organic Fluorine (AOF) in Aqueous Matrices by Combustion Ion Chromatography (CIC), January 2024. Available at: https://www.epa.gov/cwa-methods

TABLE Id–LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES[1]

Parameter	Method	EPA[2,7,10]	Standard Methods[15]	ASTM	Other
*	*	*	*	*	* *
53. Parathion ethyl	GC	614	6630 B-2021		See footnote [4], page 27; See footnote [3], p. 25.
	GC/MS	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.

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[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.

* * * * *

[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.

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[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 Ii–LIST OF APPROVED TEST PROCEDURES FOR POLYCHLORINATED BIPHENYL (PCB) CONGENERS

Parameter[1]	Congener Number[2]	Methodology	EPA	Standard Methods	ASTM	Other
1. 2-Monochlorobiphenyl	PCB-1	GC/MS	1628			
2. 3-Monochlorobiphenyl	PCB-2	GC/MS	1628			

Parameter[1]	Congener Number[2]	Methodology	EPA	Standard Methods	ASTM	Other
3. 4-Monochlorobiphenyl	PCB-3	GC/MS	1628			
4. 2,2'-Dichlorobiphenyl	PCB-4	GC/MS	1628			
5. 2,3-Dichlorobiphenyl	PCB-5	GC/MS	1628			
6. 2,3'-Dichlorobiphenyl	PCB-6	GC/MS	1628			
7. 2,4-Dichlorobiphenyl	PCB-7	GC/MS	1628			
8. 2,4'-Dichlorobiphenyl	PCB-8	GC/MS	1628			
9. 2,5-Dichlorobiphenyl	PCB-9	GC/MS	1628			
10. 2,6-Dichlorobiphenyl	PCB-10	GC/MS	1628			
11. 3,3'-Dichlorobiphenyl	PCB-11	GC/MS	1628			
12. 3,4-Dichlorobiphenyl	PCB-12	GC/MS	1628			
13. 3,4'-Dichlorobiphenyl	PCB-13	GC/MS	1628			
14. 3,5-Dichlorobiphenyl	PCB-14	GC/MS	1628			
15. 4,4'-Dichlorobiphenyl	PCB-15	GC/MS	1628			
16. 2,2',3-Trichlorobiphenyl	PCB-16	GC/MS	1628			
17. 2,2',4-Trichlorobiphenyl	PCB-17	GC/MS	1628			
18. 2,2',5-Trichlorobiphenyl	PCB-18	GC/MS	1628			
19. 2,2',6-Trichlorobiphenyl	PCB-19	GC/MS	1628			
20. 2,3,3'-Trichlorobiphenyl	PCB-20	GC/MS	1628			
21. 2,3,4-Trichlorobiphenyl	PCB-21	GC/MS	1628			
22. 2,3,4'-Trichlorobiphenyl	PCB-22	GC/MS	1628			
23. 2,3,5-Trichlorobiphenyl	PCB-23	GC/MS	1628			
24. 2,3,6-Trichlorobiphenyl	PCB-24	GC/MS	1628			
25. 2,3',4-Trichlorobiphenyl	PCB-25	GC/MS	1628			
26. 2,3',5-Trichlorobiphenyl	PCB-26	GC/MS	1628			
27. 2,3',6-Trichlorobiphenyl	PCB-27	GC/MS	1628			
28. 2,4,4'-Trichlorobiphenyl	PCB-28	GC/MS	1628			
29. 2,4,5-Trichlorobiphenyl	PCB-29	GC/MS	1628			
30. 2,4,6-Trichlorobiphenyl	PCB-30	GC/MS	1628			
31. 2,4',5-Trichlorobiphenyl	PCB-31	GC/MS	1628			
32. 2,4',6-Trichlorobiphenyl	PCB-32	GC/MS	1628			
33. 2',3,4-Trichlorobiphenyl	PCB-33	GC/MS	1628			
34. 2',3,5-Trichlorobiphenyl	PCB-34	GC/MS	1628			
35. 3,3',4-Trichlorobiphenyl	PCB-35	GC/MS	1628			
36. 3,3',5-Trichlorobiphenyl	PCB-36	GC/MS	1628			
37. 3,4,4'-Trichlorobiphenyl	PCB-37	GC/MS	1628			
38. 3,4,5-Trichlorobiphenyl	PCB-38	GC/MS	1628			
39. 3,4',5-Trichlorobiphenyl	PCB-39	GC/MS	1628			
40. 2,2',3,3'-Tetrachlorobiphenyl	PCB-40	GC/MS	1628			
41. 2,2',3,4-Tetrachlorobiphenyl	PCB-41	GC/MS	1628			
42. 2,2',3,4'-Tetrachlorobiphenyl	PCB-42	GC/MS	1628			
43. 2,2',3,5-Tetrachlorobiphenyl	PCB-43	GC/MS	1628			
44. 2,2',3,5'-Tetrachlorobiphenyl	PCB-44	GC/MS	1628			
45. 2,2',3,6-Tetrachlorobiphenyl	PCB-45	GC/MS	1628			
46. 2,2',3,6'-Tetrachlorobiphenyl	PCB-46	GC/MS	1628			
47. 2,2',4,4'-Tetrachlorobiphenyl	PCB-47	GC/MS	1628			
48. 2,2',4,5-Tetrachlorobiphenyl	PCB-48	GC/MS	1628			
49. 2,2',4,5'-Tetrachlorobiphenyl	PCB-49	GC/MS	1628			
50. 2,2',4,6-Tetrachlorobiphenyl	PCB-50	GC/MS	1628			
51. 2,2',4,6'-Tetrachlorobiphenyl	PCB-51	GC/MS	1628			
52. 2,2',5,5'-Tetrachlorobiphenyl	PCB-52	GC/MS	1628			
53. 2,2',5,6'-Tetrachlorobiphenyl	PCB-53	GC/MS	1628			
54. 2,2',6,6'-Tetrachlorobiphenyl	PCB-54	GC/MS	1628			
55. 2,3,3',4-Tetrachlorobiphenyl	PCB-55	GC/MS	1628			
56. 2,3,3',4'-Tetrachlorobiphenyl	PCB-56	GC/MS	1628			
57. 2,3,3',5-Tetrachlorobiphenyl	PCB-57	GC/MS	1628			
58. 2,3,3',5'-Tetrachlorobiphenyl	PCB-58	GC/MS	1628			

Parameter[1]	Congener Number[2]	Methodology	EPA	Standard Methods	ASTM	Other
59. 2,3,3',6-Tetrachlorobiphenyl	PCB-59	GC/MS	1628			
60. 2,3,4,4'-Tetrachlorobiphenyl	PCB-60	GC/MS	1628			
61. 2,3,4,5-Tetrachlorobiphenyl	PCB-61	GC/MS	1628			
62. 2,3,4,6-Tetrachlorobiphenyl	PCB-62	GC/MS	1628			
63. 2,3,4',5-Tetrachlorobiphenyl	PCB-63	GC/MS	1628			
64. 2,3,4',6-Tetrachlorobiphenyl	PCB-64	GC/MS	1628			
65. 2,3,5,6-Tetrachlorobiphenyl	PCB-65	GC/MS	1628			
66. 2,3',4,4'-Tetrachlorobiphenyl	PCB-66	GC/MS	1628			
67. 2,3',4,5-Tetrachlorobiphenyl	PCB-67	GC/MS	1628			
68. 2,3',4,5'-Tetrachlorobiphenyl	PCB-68	GC/MS	1628			
69. 2,3',4,6-Tetrachlorobiphenyl	PCB-69	GC/MS	1628			
70. 2,3',4',5-Tetrachlorobiphenyl	PCB-70	GC/MS	1628			
71. 2,3',4',6-Tetrachlorobiphenyl	PCB-71	GC/MS	1628			
72. 2,3',5,5'-Tetrachlorobiphenyl	PCB-72	GC/MS	1628			
73. 2,3',5',6-Tetrachlorobiphenyl	PCB-73	GC/MS	1628			
74. 2,4,4',5-Tetrachlorobiphenyl	PCB-74	GC/MS	1628			
75. 2,4,4',6-Tetrachlorobiphenyl	PCB-75	GC/MS	1628			
76. 2',3,4,5-Tetrachlorobiphenyl	PCB-76	GC/MS	1628			
77. 3,3',4,4'-Tetrachlorobiphenyl	PCB-77	GC/MS	1628			
78. 3,3',4,5-Tetrachlorobiphenyl	PCB-78	GC/MS	1628			
79. 3,3',4,5'-Tetrachlorobiphenyl	PCB-79	GC/MS	1628			
80. 3,3',5,5'-Tetrachlorobiphenyl	PCB-80	GC/MS	1628			
81. 3,4,4',5-Tetrachlorobiphenyl	PCB-81	GC/MS	1628			
82. 2,2',3,3',4-Pentachlorobiphenyl	PCB-82	GC/MS	1628			
83. 2,2',3,3',5-Pentachlorobiphenyl	PCB-83	GC/MS	1628			
84. 2,2',3,3',6-Pentachlorobiphenyl	PCB-84	GC/MS	1628			
85. 2,2',3,4,4'-Pentachlorobiphenyl	PCB-85	GC/MS	1628			
86. 2,2',3,4,5-Pentachlorobiphenyl	PCB-86	GC/MS	1628			
87. 2,2',3,4,5'-Pentachlorobiphenyl	PCB-87	GC/MS	1628			
88. 2,2',3,4,6-Pentachlorobiphenyl	PCB-88	GC/MS	1628			
89. 2,2',3,4,6'-Pentachlorobiphenyl	PCB-89	GC/MS	1628			
90. 2,2',3,4',5-Pentachlorobiphenyl	PCB-90	GC/MS	1628			
91. 2,2',3,4',6-Pentachlorobiphenyl	PCB-91	GC/MS	1628			
92. 2,2',3,5,5'-Pentachlorobiphenyl	PCB-92	GC/MS	1628			
93. 2,2',3,5,6-Pentachlorobiphenyl	PCB-93	GC/MS	1628			
94. 2,2',3,5,6'-Pentachlorobiphenyl	PCB-94	GC/MS	1628			
95. 2,2',3,5',6-Pentachlorobiphenyl	PCB-95	GC/MS	1628			
96. 2,2',3,6,6'-Pentachlorobiphenyl	PCB-96	GC/MS	1628			
97. 2,2',3',4,5-Pentachlorobiphenyl	PCB-97	GC/MS	1628			
98. 2,2',3',4,6-Pentachlorobiphenyl	PCB-98	GC/MS	1628			
99. 2,2',4,4',5-Pentachlorobiphenyl	PCB-99	GC/MS	1628			
100. 2,2',4,4',6-Pentachlorobiphenyl	PCB-100	GC/MS	1628			
101. 2,2',4,5,5'-Pentachlorobiphenyl	PCB-101	GC/MS	1628			
102. 2,2',4,5,6'-Pentachlorobiphenyl	PCB-102	GC/MS	1628			
103. 2,2',4,5,'6-Pentachlorobiphenyl	PCB-103	GC/MS	1628			
104. 2,2',4,6,6'-Pentachlorobiphenyl	PCB-104	GC/MS	1628			
105. 2,3,3',4,4'-Pentachlorobiphenyl	PCB-105	GC/MS	1628			
106. 2,3,3',4,5-Pentachlorobiphenyl	PCB-106	GC/MS	1628			
107. 2,3,3',4',5-Pentachlorobiphenyl	PCB-107	GC/MS	1628			
108. 2,3,3',4,5'-Pentachlorobiphenyl	PCB-108	GC/MS	1628			
109. 2,3,3',4,6-Pentachlorobiphenyl	PCB-109	GC/MS	1628			
110. 2,3,3',4',6-Pentachlorobiphenyl	PCB-110	GC/MS	1628			
111. 2,3,3',5,5'-Pentachlorobiphenyl	PCB-111	GC/MS	1628			
112. 2,3,3',5,6-Pentachlorobiphenyl	PCB-112	GC/MS	1628			
113. 2,3,3',5',6-Pentachlorobiphenyl	PCB-113	GC/MS	1628			
114. 2,3,4,4',5-Pentachlorobiphenyl	PCB-114	GC/MS	1628			

Parameter[1]	Congener Number[2]	Methodology	EPA	Standard Methods	ASTM	Other
115. 2,3,4,4',6-Pentachlorobiphenyl	PCB-115	GC/MS	1628			
116. 2,3,4,5,6-Pentachlorobiphenyl	PCB-116	GC/MS	1628			
117. 2,3,4',5,6-Pentachlorobiphenyl	PCB-117	GC/MS	1628			
118. 2,3',4,4',5-Pentachlorobiphenyl	PCB-118	GC/MS	1628			
119. 2,3',4,4',6-Pentachlorobiphenyl	PCB-119	GC/MS	1628			
120. 2,3',4,5,5'-Pentachlorobiphenyl	PCB-120	GC/MS	1628			
121. 2,3',4,5,'6-Pentachlorobiphenyl	PCB-121	GC/MS	1628			
122. 2',3,3',4,5-Pentachlorobiphenyl	PCB-122	GC/MS	1628			
123. 2',3,4,4',5-Pentachlorobiphenyl	PCB-123	GC/MS	1628			
124. 2',3,4,5,5'-Pentachlorobiphenyl	PCB-124	GC/MS	1628			
125. 2',3,4,5,6'-Pentachlorobiphenyl	PCB-125	GC/MS	1628			
126. 3,3',4,4',5-Pentachlorobiphenyl	PCB-126	GC/MS	1628			
127. 3,3',4,5,5'-Pentachlorobiphenyl	PCB-127	GC/MS	1628			
128. 2,2',3,3',4,4'-Hexachlorobiphenyl	PCB-128	GC/MS	1628			
129. 2,2',3,3',4,5-Hexachlorobiphenyl	PCB-129	GC/MS	1628			
130. 2,2',3,3',4,5'-Hexachlorobiphenyl	PCB-130	GC/MS	1628			
131. 2,2',3,3',4,6-Hexachlorobiphenyl	PCB-131	GC/MS	1628			
132. 2,2',3,3',4,6'-Hexachlorobiphenyl	PCB-132	GC/MS	1628			
133. 2,2',3,3',5,5'-Hexachlorobiphenyl	PCB-133	GC/MS	1628			
134. 2,2',3,3',5,6-Hexachlorobiphenyl	PCB-134	GC/MS	1628			
135. 2,2',3,3',5,6'-Hexachlorobiphenyl	PCB-135	GC/MS	1628			
136. 2,2',3,3',6,6'-Hexachlorobiphenyl	PCB-136	GC/MS	1628			
137. 2,2',3,4,4',5-Hexachlorobiphenyl	PCB-137	GC/MS	1628			
138. 2,2',3,4,4',5'-Hexachlorobiphenyl	PCB-138	GC/MS	1628			
139. 2,2',3,4,4',6-Hexachlorobiphenyl	PCB-139	GC/MS	1628			
140. 2,2',3,4,4',6'-Hexachlorobiphenyl	PCB-140	GC/MS	1628			
141. 2,2',3,4,5,5'-Hexachlorobiphenyl	PCB-141	GC/MS	1628			
142. 2,2',3,4,5,6-Hexachlorobiphenyl	PCB-142	GC/MS	1628			
143. 2,2',3,4,5,6'-Hexachlorobiphenyl	PCB-143	GC/MS	1628			
144. 2,2',3,4,5',6-Hexachlorobiphenyl	PCB-144	GC/MS	1628			
145. 2,2',3,4,6,6'-Hexachlorobiphenyl	PCB-145	GC/MS	1628			
146. 2,2',3,4',5,5'-Hexachlorobiphenyl	PCB-146	GC/MS	1628			
147. 2,2',3,4',5,6-Hexachlorobiphenyl	PCB-147	GC/MS	1628			
148. 2,2',3,4',5,6'-Hexachlorobiphenyl	PCB-148	GC/MS	1628			
149. 2,2',3,4',5',6-Hexachlorobiphenyl	PCB-149	GC/MS	1628			
150. 2,2',3,4',6,6'-Hexachlorobiphenyl	PCB-150	GC/MS	1628			
151. 2,2',3,5,5',6-Hexachlorobiphenyl	PCB-151	GC/MS	1628			
152. 2,2',3,5,6,6'-Hexachlorobiphenyl	PCB-152	GC/MS	1628			
153. 2,2',4,4',5,5'-Hexachlorobiphenyl	PCB-153	GC/MS	1628			
154. 2,2',4,4',5,6’-Hexachlorobiphenyl	PCB-154	GC/MS	1628			
155. 2,2',4,4',6,6'-Hexachlorobiphenyl	PCB-155	GC/MS	1628			
156. 2,3,3',4,4',5-Hexachlorobiphenyl	PCB-156	GC/MS	1628			
157. 2,3,3',4,4',5'-Hexachlorobiphenyl	PCB-157	GC/MS	1628			
158. 2,3,3',4,4',6-Hexachlorobiphenyl	PCB-158	GC/MS	1628			
159. 2,3,3',4,5,5'-Hexachlorobiphenyl	PCB-159	GC/MS	1628			
160. 2,3,3',4,5,6-Hexachlorobiphenyl	PCB-160	GC/MS	1628			
161. 2,3,3',4,5',6-Hexachlorobiphenyl	PCB-161	GC/MS	1628			
162. 2,3,3',4',5,5'-Hexachlorobiphenyl	PCB-162	GC/MS	1628			
163. 2,3,3',4',5,6-Hexachlorobiphenyl	PCB-163	GC/MS	1628			
164. 2,3,3',4',5',6-Hexachlorobiphenyl	PCB-164	GC/MS	1628			
165. 2,3,3',5,5',6-Hexachlorobiphenyl	PCB-165	GC/MS	1628			
166. 2,3,4,4',5,6-Hexachlorobiphenyl	PCB-166	GC/MS	1628			
167. 2,3',4,4',5,5'-Hexachlorobiphenyl	PCB-167	GC/MS	1628			
168. 2,3',4,4',5',6-Hexachlorobiphenyl	PCB-168	GC/MS	1628			
169. 3,3',4,4',5,5'-Hexachlorobiphenyl	PCB-169	GC/MS	1628			
170. 2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB-170	GC/MS	1628			

Parameter[1]	Congener Number[2]	Methodology	EPA	Standard Methods	ASTM	Other
171. 2,2'3,3',4,4',6-Heptachlorobiphenyl	PCB-171	GC/MS	1628			
172. 2,2',3,3',4,5,5'-Heptachlorobiphenyl	PCB-172	GC/MS	1628			
173. 2,2',3,3',4,5,6-Heptachlorobiphenyl	PCB-173	GC/MS	1628			
174. 2,2',3,3',4,5,6'-Heptachlorobiphenyl	PCB-174	GC/MS	1628			
175. 2,2',3,3',4,5',6-Heptachlorobiphenyl	PCB-175	GC/MS	1628			
176. 2,2',3,3',4,6,6'-Heptachlorobiphenyl	PCB-176	GC/MS	1628			
177. 2,2',3,3',4',5,6-Heptachlorobiphenyl	PCB-177	GC/MS	1628			
178. 2,2',3,3',5,5',6-Heptachlorobiphenyl	PCB-178	GC/MS	1628			
179. 2,2',3,3',5,6,6'-Heptachlorobiphenyl	PCB-179	GC/MS	1628			
180. 2,2',3,4,4',5,5'-Heptachlorobiphenyl	PCB-180	GC/MS	1628			
181. 2,2',3,4,4',5,6-Heptachlorobiphenyl	PCB-181	GC/MS	1628			
182. 2,2',3,4,4',5,6'-Heptachlorobiphenyl	PCB-182	GC/MS	1628			
183. 2,2',3,4,4',5',6-Heptachlorobiphenyl	PCB-183	GC/MS	1628			
184. 2,2',3,4,4',6,6'-Heptachlorobiphenyl	PCB-184	GC/MS	1628			
185. 2,2',3,4,5,5',6-Heptachlorobiphenyl	PCB-185	GC/MS	1628			
186. 2,2',3,4,5,6,6'-Heptachlorobiphenyl	PCB-186	GC/MS	1628			
187. 2,2',3,4',5,5',6-Heptachlorobiphenyl	PCB-187	GC/MS	1628			
188. 2,2',3,4',5,6,6'-Heptachlorobiphenyl	PCB-188	GC/MS	1628			
189. 2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB-189	GC/MS	1628			
190. 2,3,3',4,4',5,6-Heptachlorobiphenyl	PCB-190	GC/MS	1628			
191. 2,3,3',4,4',5',6-Heptachlorobiphenyl	PCB-191	GC/MS	1628			
192. 2,3,3',4,5,5',6-Heptachlorobiphenyl	PCB-192	GC/MS	1628			
193. 2,3,3',4',5,5',6-Heptachlorobiphenyl	PCB-193	GC/MS	1628			
194. 2,2',3,3',4,4',5,5'-Octachlorobiphenyl	PCB-194	GC/MS	1628			
195. 2,2',3,3',4,4',5,6-Octachlorobiphenyl	PCB-195	GC/MS	1628			
196. 2,2',3,3',4,4',5,6'-Octachlorobiphenyl	PCB-196	GC/MS	1628			
197. 2,2',3,3',4,4',6,6'-Octachlorobiphenyl	PCB-197	GC/MS	1628			
198. 2,2',3,3',4,5,5',6-Octachlorobiphenyl	PCB-198	GC/MS	1628			
199. 2,2',3,3',4,5,5',6'-Octachlorobiphenyl	PCB-199	GC/MS	1628			
200. 2,2',3,3',4,5,6,6'-Octachlorobiphenyl	PCB-200	GC/MS	1628			
201. 2,2',3,3',4,5',6,6'-Octachlorobiphenyl	PCB-201	GC/MS	1628			
202. 2,2',3,3',5,5',6,6'-Octachlorobiphenyl	PCB-202	GC/MS	1628			
203. 2,2',3,4,4',5,5',6-Octachlorobiphenyl	PCB-203	GC/MS	1628			
204. 2,2',3,4,4',5,6,6'-Octachlorobiphenyl	PCB-204	GC/MS	1628			
205. 2,3,3',4,4',5,5',6-Octachlorobiphenyl	PCB-205	GC/MS	1628			
206. 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB-206	GC/MS	1628			
207. 2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	PCB-207	GC/MS	1628			
208. 2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	PCB-208	GC/MS	1628			
209. Decachlorobiphenyl	PCB-209	GC/MS	1628			
210. Total PCBs[3]	Total PCBs	GC/MS	1628			

Table Ii notes:

[1] EPA Substance Registry Services name (https://cdxapps.epa.gov/oms-substance-registry-services/)

[2] Preferred abbreviation to facilitate consistency in reporting of the results for these parameters.

[3] Total PCBs is the sum of all the detected *c*ongeners in the sample. Calculate the total PCB concentration by using zero (0) for the contribution of the congeners that are not detected. Otherwise, every method blank will have a non-zero result, even when no congeners are detected, affecting assessments of the sample results relative to the method blank. Further, there is no method detection limit (MDL) for Total PCBs

because it is the sum of the results for many individual parameters. Method blanks and samples with no detects are reported as "ND" for non-detect for the Total PCBs result (or a similarly appropriate non-numeric value).

TABLE IJ–LIST OF APPROVED TEST PROCEDURES FOR PER- AND POLYFLUORINATED ALKYL SUBSTANCES (PFAS)

Parameter[1]	Abbreviation[2]	Methodology	EPA	Standard Methods	ASTM[3]	Other
1. Perfluorobutanoic acid	PFBA	LC-MS/MS	1633A		D8421-24	
2. Perfluoropentanoic acid	PFPeA	LC-MS/MS	1633A		D8421-24	
3. Perfluorohexanoic acid	PFHxA	LC-MS/MS	1633A		D8421-24	
4. Perfluoroheptanoic acid	PFHpA	LC-MS/MS	1633A		D8421-24	
5. Perfluorooctanoic acid	PFOA	LC-MS/MS	1633A		D8421-24	
6. Perfluorononanoic acid	PFNA	LC-MS/MS	1633A		D8421-24	
7. Perfluorodecanoic acid	PFDA	LC-MS/MS	1633A		D8421-24	
8. Perfluoroundecanoic acid	PFUnA	LC-MS/MS	1633A		D8421-24	
9. Perfluorododecanoic acid	PFDoA	LC-MS/MS	1633A		D8421-24	
10. Perfluorotridecanoic acid	PFTrDA	LC-MS/MS	1633A		D8421-24	
11. Perfluorotetradecanoic acid	PFTeDA	LC-MS/MS	1633A		D8421-24	
12. Perfluorobutanesulfonic acid	PFBS	LC-MS/MS	1633A		D8421-24	
13. Perfluoropentanesulfonic acid	PFPeS	LC-MS/MS	1633A		D8421-24	
14. Perfluorohexanesulfonic acid	PFHxS	LC-MS/MS	1633A		D8421-24	
15. Perfluoroheptanesulfonic acid	PFHpS	LC-MS/MS	1633A		D8421-24	
16. Perfluorooctanesulfonic acid	PFOS	LC-MS/MS	1633A		D8421-24	
17. Perfluorononanesulfonic acid	PFNS	LC-MS/MS	1633A		D8421-24	
18. Perfluorodecanesulfonic acid	PFDS	LC-MS/MS	1633A		D8421-24	
19. Perfluorododecanesulfonic acid	PFDoS	LC-MS/MS	1633A		D8421-24	
20. 1H,1H,2H,2H-Perfluorohexane sulfonic acid	4:2FTS	LC-MS/MS	1633A		D8421-24	
21. 1H,1H,2H,2H-Perfluorooctane sulfonic acid	6:2FTS	LC-MS/MS	1633A		D8421-24	
22. 1H,1H,2H,2H-Perfluorodecane sulfonic acid	8:2FTS	LC-MS/MS	1633A		D8421-24	
23. Perfluorooctanesulfonamide	PFOSA	LC-MS/MS	1633A		D8421-24	
24. N-methyl perfluorooctanesulfonamide	NMeFOSA	LC-MS/MS	1633A		D8421-24	
25. N-ethyl perfluorooctanesulfonamide	NEtFOSA	LC-MS/MS	1633A		D8421-24	
26. N-methyl perfluorooctanesulfonamido-acetic acid	NMeFOSAA	LC-MS/MS	1633A		D8421-24	
27. N-ethyl perfluorooctanesulfonamido-acetic acid	NEtFOSAA	LC-MS/MS	1633A		D8421-24	
28. N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	LC-MS/MS	1633A		D8421-24	
29. N-ethyl perfluorooctanesulfonamido-ethanol	NEtFOSE	LC-MS/MS	1633A		D8421-24	
30. Hexafluoropropylene oxide dimer acid	HFPO-DA	LC-MS/MS	1633A		D8421-24	
31. 4,8-Dioxa-3H-perfluorononanoic acid	ADONA	LC-MS/MS	1633A		D8421-24	
32. Perfluoro-3-methoxypropanoic acid	PFMPA	LC-MS/MS	1633A		D8421-24	
33. Perfluoro-4-methoxybutanoic acid	PFMBA	LC-MS/MS	1633A		D8421-24	
34. Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	LC-MS/MS	1633A		D8421-24	
35. 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	LC-MS/MS	1633A		D8421-24	
36. 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	LC-MS/MS	1633A		D8421-24	

Parameter[1]	Abbreviation[2]	Methodology	EPA	Standard Methods	ASTM[3]	Other
37. Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	LC-MS/MS	1633A		D8421-24	
38. 3-Perfluoropropyl propanoic acid	3:3FTCA	LC-MS/MS	1633A		D8421-24	
39. 2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA	LC-MS/MS	1633A		D8421-24	
40. 3-Perfluoroheptyl propanoic acid	7:3FTCA	LC-MS/MS	1633A		D8421-24	

Table Ij notes:

[1] EPA Substance Registry Services name (https://cdxapps.epa.gov/oms-substance-registry-services/)

[2] Preferred Abbreviation from Method 1633 to facilitate consistency in reporting of the results for these parameters.

[3] ASTM D8421-24 is only applicable to aqueous samples.

(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 incorporation by reference (IBR) 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/ibrlocations or email fr.inspection@nara.gov*. The material may be obtained from the following sources in this paragraph (b).

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(xi) Method 1621, Determination of Adsorbable Organic Fluorine (AOF) in Aqueous Matrices by Combustion Ion Chromatography (CIC). EPA 821-R-24-002. Table Ic.

(xii) Method 1628 Method 1628 Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-resolution GC/MS using Selected Ion Monitoring. July 2021. EPA 821-R-21-002. Table Ii.

(xiii) Method 1633A Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. EPA-820-R-24-007. Table Ij.

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(10) American Public Health Association, 800 I Street, NW, Washington, DC 20001; phone: 202-777-2742; website: *www.standardmethods.org*.

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

(ii) Standard Methods for the Examination of Water and Wastewater. 15th Edition, 1980, Table IB, Note 30; Table ID.

(iii) 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. Table IC, Note 6; Table ID, Note 6.

(iv) Standard Methods for the Examination of Water and Wastewater. 18th Edition, 1992. Tables IA, IB, IC, ID, IE, and IH.

(v) Standard Methods for the Examination of Water and Wastewater. 19th Edition, 1995. Tables IA, IB, IC, ID, IE, and IH.

(vi) Standard Methods for the Examination of Water and Wastewater. 20th Edition, 1998. Tables IA, IB, IC, ID, IE, and IH.

(vii) Standard Methods for the Examination of Water and Wastewater. 21st Edition, 2005. Table IB, Notes 17 and 27.

(viii) 2120, Color. Revised September 4, 2021. Table IB.

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

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

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

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

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

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

(xv) 2550, Temperature. 2010. Table IB.

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

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

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

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

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

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

(xxii) 3500-Ca, Calcium. Revised 2020. Table IB.

(xxiii) 3500-Cr, Chromium. Revised 2020. Table IB.

(xxiv) 3500-K, Potassium. Revised 2020. Table IB.

(xxv) 3500-Na, Sodium. Revised 2020. Table IB.

(xxvi) 4500-H2O2 Hydrogen Peroxide (Residual) 2020. Table Ib.

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

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

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

(xxx) 4500-Cl, Chlorine (Residual). 2011. Table IB.

(xxxi) 4500-CN^-, Cyanide. Revised 2021. Table IB.

(xxxii) 4500-PAA Peracetic Acid (Residual) 2019. Table Ib.

(xxxiii) 4500-F^-, Fluoride. Revised 2021. Table IB.

(xxxiv) 4500-H^+, pH. 2021. Table IB.

(xxxv) 4500-NH_3, Nitrogen (Ammonia). Revised 2021. Table IB.

(xxxvi) 4500-NO_2^-, Nitrogen (Nitrite). Revised 2021. Table IB.

(xxxvii) 4500-NO_3^-, Nitrogen (Nitrate). Revised 2019. Table IB.

(xxxviii) 4500-$N_{(org)}$, Nitrogen (Organic). Revised 2021. Table IB.

(xxxix) 4500-O, Oxygen (Dissolved). Revised 2021. Table IB.

(xl) 4500-P, Phosphorus. Revised 2021. Table IB.

(xli) 4500-SiO_2, Silica. Revised 2021. Table IB.

(xlii) 4500-S^{2-}, Sulfide. Revised 2021. Table IB.

(xliii) 4500-SO_3^{2-}, Sulfite. Revised 2021. Table IB.

(xliv) 4500-SO_4^{2-}, Sulfate. Revised 2021. Table IB.

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

(xlvi) 5220, Chemical Oxygen Demand (COD). 2011. Table IB.

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

(xlviii) 5520, Oil and Grease. Revised 2021. Table IB.

(xlix) 5530, Phenols. Revised 2021. Table IB.

(l) 5540, Surfactants. Revised 2021. Table IB.

(li) 6200, Volatile Organic Compounds. Revised 2020. Table IC.

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

(liii) 6420, Phenols. Revised 2021. Table IC.

(liv) 6440, Polynuclear Aromatic Hydrocarbons. Revised 2021. Table IC.

(lv) 6630, Organochlorine Pesticides. Revised 2021. Table ID.

(lvi) 6640, Acidic Herbicide Compounds. Revised 2021. Table ID.

(lvii) 7110, Gross Alpha and Gross Beta Radioactivity (Total, Suspended, and Dissolved). 2000. Table IE.

(lviii) 7500, Radium. 2001. Table IE.

(lix) 9213, Recreational Waters. 2007. Table IH.

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

(lxi) 9222, Membrane Filter Technique for Members of the Coliform Group. 2015. Table IA, Note 31; Table IH, Note 17.

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

(lxiii) 9230 Fecal Enterococcus/Streptococcus Groups. 2013. Table IA, Note 32; Table IH.

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(lxxi) ASTM D8421-24, Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices by Co-solvation followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS). June 2024. Table Ij.

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(i) Organochlorine Pesticides and PCBs in Wastewater Using EmporeTM Disk” Test Method 3M 0222. Revised October 28, 1994. Table Id, Note 8.

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

Parameter number/name	Container [1]	Preservation [2,3]	Maximum holding time [4]
* * * * * * *			
Table Ib—Inorganic Tests			
* * * * * * *			
4. Ammonia	P, FP, G	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], H_2SO_4 to pH <2	28 days
* * * * * * *			
48. Phenols	G	Cool, ≤6 °C[18], , 0.008% $Na_2S_2O_3$[5], $H_2 SO_4$ to pH <2	28 days.
* * * * * * *			
50. Phosphorus, total	P, FP,G	Cool, ≤6 °C 18, H2SO4 to pH <2 [25]	28 days.
* * * * * * *			
77. Hydrogen Peroxide	P, G	None required	Analyze within 15 minutes.
78. Peracetic Acid	P, G	None required	Analyze within 15 minutes.
* * * * * * *			
Table Ic—Organic Tests [8]			
13, 18-20, 22, 24, 25, 27, 28, 34-37, 39-43, 45-47, 56, 76, 97, 98, 101-104, 106. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9]	14 days [9]
26. 2-Chloroethylvinyl ether	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2 S_2 O_3$[5]	14 days[9]
6, 57, 99. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9]	14 days [9]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$, HCl to pH 2[10]	14 days [10]

Parameter number/name	Container [1]	Preservation [2,3]	Maximum holding time [4]
23, 30, 44, 49, 53, 77, 80, 81, 91, 93, 105. Phenols [11]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
7, 38. Benzidines[11 12]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2 S_2 O3$[5]	7 days until extraction.[13]
14, 17, 48, 50-52. Phthalate esters[11]	G, FP-lined cap	Cool, ≤6 °C[18]	7 days until extraction, 40 days after extraction
82-84. Nitrosamines[11 14]	G, FP-lined cap	Cool, ≤6 °C[18], store in dark, 0.008% $Na_2 S_2 O_3$[5]	7 days until extraction, 40 days after extraction.
88-94. PCBs[11]	G, FP-lined cap	Cool, ≤6 °C[18]	1 year until extraction, 1 year after extraction.
54, 55, 75, 79 Nitroaromatics and isophorone[11]	G, FP-lined cap	Cool, ≤6 °C[18], store in dark, 0.008% $Na_2 S_2 O_3$[5]	7 days until extraction, 40 days after extraction.
1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 92, 94. Polynuclear aromatic hydrocarbons [11]	G, FP-lined cap	Cool, ≤6 °C[18], store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 87. Haloethers[11]	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2 S_2 O_3$[5]	7 days until extraction, 40 days after extraction.
29, 35-37, 63-65, 73, 100. Chlorinated hydrocarbons [11]	G, FP-lined cap	Cool, ≤6 °C[18]	7 days until extraction, 40 days after extraction
60-62, 66-72, 85, 86, 88-90, 95, 96. CDDs/CDFs [11]	G	See footnote 11	See footnote 11
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C[18], 0.008% $Na_2 S_2 O_3$[5], pH <9	1 Year.
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C[18]	7 Days.
Tissue Samples: Field Preservation	G	Cool, ≤6 °C[18]	24 Hours

Parameter number/name	Container [1]	Preservation [2,3]	Maximum holding time [4]
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤−10 °C	1 Year
107-111. Alkylated phenols	G	Cool, <6 °C, 0.008% $Na_2S_2O_3$[5], H_2SO_4 to pH <2	28 days until extraction, 40 days after extraction
112. Adsorbable Organic Halides (AOX)	G	Cool, <6 °C, 0.008% $Na_2S_2O_3$[5], HNO_3 to pH <2	Hold *at least* 3 days, but not more than 6 months
113. Chlorinated Phenolics	G, FP-lined cap	Cool, <6 °C, 0.008% $Na_2S_2O_3$[5], H_2SO_4 to pH <2	30 days until acetylation, 30 days after acetylation
114. Adsorbable Organic Fluorine (AOF)	HDPE with linerless HDPE or PP caps [26]	Cool, <6 °C	90 days
* * * * * * *			
Table Ii—Polychlorinated Biphenyl Congener Tests			
1-210. PCB Congeners			
Aqueous Samples: Field Preservation	G, FP-lined cap	Cool, ≤6 °C[18], 0.008% $Na_2S_2O_3$[5]	1 year
Solids, Biosolids, and Mixed-Phase Samples: Field Preservation	G, FP-lined cap	Cool, ≤6 °C[18]	1 year
Tissue Samples: Field Preservation	G, FP-lined cap	Cool, ≤6 °C[18]	24 hours
Solids, Biosolids, Mixed-Phase, and Tissue Samples: Lab Preservation	G, FP-lined cap	Freeze, ≤ -10 °C	1 year
Sample Extracts	G, amber	Freeze, < -20 °C	1 year
Table Ij—Per- and Polyfluorinated Alkyl Substances Tests			
1-40. PFAS			
Aqueous Samples: Field Preservation	HDPE or PP, linerless cap[27]	Cool, ≤6 °C[18]	48 hours
Solids, Biosolids, and Mixed-Phase Samples: Field Preservation	HDPE, linerless cap[27]	Cool, ≤6 °C[18]	24 hours [28], or 48 hours
Tissue Samples: Field Preservation	HDPE, linerless cap[27]	Cool, ≤6 °C[18], or Freeze, ≤ -20 °C	24 hours [28], or 48 hours [29]

Parameter number/name	Container [1]	Preservation [2,3]	Maximum holding time [4]
Leachate Samples: Field Preservation	HDPE or PP, linerless cap[27]	Cool, ≤6 °C[18]	48 hours
Aqueous and Leachate Samples: Lab Preservation	HDPE or PP, linerless cap[27]	Cool, ≤6 °C[18], or Freeze, ≤ -20 °C	28 days, or 90 days
Solids, Biosolids, and Mixed-Phase Samples: Lab Preservation	HDPE, linerless cap[27]	Cool, ≤6 °C[18], or Freeze, ≤ -20 °C	90 days
Tissue Samples: Lab Preservation	HDPE, linerless cap[27]	Freeze, ≤ -20 °C	90 days
Sample Extracts		Cool, ≤6 °C[18], or Freeze, ≤ 0 °C	90 days

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

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

procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.24(f)(14)(iv) and (v) (volatile organics).

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

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

automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0-6 °C, with minimum head space.

[5] ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g., chlorine) for cyanide analyses. Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (23rd edition) addresses dechlorination procedures for microbiological analyses. Section A.2 of Standard Method 4500-NH3 discusses the need for dechlorination of samples for ammonia analysis. Addition of sodium thiosulfate to samples for other parameters is only necessary when oxidants are present (e.g., chlorine).

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

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

[10] Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

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

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

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

[14] For the analysis of diphenylnitrosamine, add 0.008% Na2 S2 O3 and adjust pH to 7-10 with NaOH within 24 hours of sampling.

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[17] Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 or EPA Method 245.7 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[18] Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ 6 °C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤ 6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

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[25] Samples to be analyzed for total phosphorous using EPA Method 200.7 or another approved ICP/AES method may be preserved using nitric acid to pH < 2, or the acid-preserved sample for metals may be used for analysis, and the holding time for total phosphorus may be extended to 6 months.

[26] For AOF, collect three separate containers (see Section 8 of Method 1621).

[27] Samples for EPA Method 1633 are collected in HDPE containers with linerless HDPE or PP caps. Aqueous samples for ASTM D841-22 are collected in pre-labeled and pre-weighed 15-mL PP tubes. Containers for either method must be lot-tested and shown to be free of detectable PFAS.

[28] Samples should be extracted within 24 hours if NFDHA is a required analyte for a given project.

[29] If the whole fish or other seafood sample is frozen within 48 hours of collection, the holding time begins when the tissue is processed for analysis (e.g., filleting, grinding, removal of specific tissue for analysis. Prepared tissue sample aliquots may be held for up to 90 days when stored at less than or equal to -20 °C.