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Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; Proposed Rule

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 136, 260, 423, 430, and 435

[EPA–HQ–OW–2010–0192; FRL–9189–4]

RIN 2040–AF09

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule.

SUMMARY: EPA is proposing changes to analysis and sampling test procedures in wastewater regulations. These changes will provide increased flexibility to the regulated community and laboratories in their selection of analytical methods (test procedures) for use in Clean Water Act programs. The changes include proposal of EPA methods and methods published by voluntary consensus standard bodies, such as ASTM International and the Standard Methods Committee and updated versions of currently approved methods. EPA is also proposing to add certain methods reviewed under the alternate test procedures program. Further, EPA is proposing changes to the current regulations to clarify the process for EPA approval for use of alternate procedures for nationwide and Regional use. In addition, EPA is proposing minimum quality control requirements to improve consistency across method versions; corrections to previously approved methods; and changes to sample collection, preservation, and holding time requirements. Finally, EPA is proposing changes to how EPA cites methods in three effluent guideline regulations.

DATES: EPA must receive your comments on this proposal on or before November 22, 2010.

ADDRESSES: Submit your comments, identified by Docket ID No. EPA–HQ–OW–2010–0192, by one of the following methods:

- *http://www.regulations.gov:* Follow the on-line instructions for submitting comments.
- *E-mail: OW-Docket@epa.gov,* Attention Docket ID No. EPA–HQ–OW–2010–0192.
- *Mail:* Water Docket, U.S. Environmental Protection Agency, Mailcode: 2822T, 1200 Pennsylvania Ave., NW., Washington, DC 20460. Attention Docket ID No. EPA–HQ–OW–2010–0192. Please include a total of 3 copies.
- *Hand Delivery:* Water Docket, EPA Docket Center, EPA West Building Room 3334, 1301 Constitution Ave., NW., Washington, DC, Attention Docket ID No. EPA–HQ–OW–2010–0192. Such deliveries are only accepted during the Docket's normal hours of operation, and special arrangements should be made for deliveries of boxed information by calling 202–566–2426.

Instructions: Direct your comments to Docket ID No. EPA–HQ–OW–2010–0192. EPA's policy is that all comments received will be included in the public docket without change and may be made available online at *http://www.regulations.gov,* including any personal information provided, unless the comment includes information claimed to be Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Do not submit information that you consider to be CBI or otherwise protected through *http://www.regulations.gov* or e-mail. The *http://www.regulations.gov* Web site is an "anonymous access" system, which means EPA will not know your identity or contact information unless you provide it in the body of your comment. If you send an e-mail comment directly to EPA without going through *http://www.regulations.gov* your e-mail address will be automatically captured and included as part of the comment that is placed in the public docket and made available on the Internet. If you submit an electronic comment, EPA recommends that you include your name and other contact information in the body of your comment and with any disk or CD–ROM you submit. If EPA cannot read your comment due to technical difficulties and cannot contact you for clarification, EPA may not be able to consider your comment. Electronic files should avoid the use of special characters, any form of encryption, and be free of any defects or viruses.

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

FOR FURTHER INFORMATION CONTACT: Lemuel Walker, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave., NW., Washington, DC 20460, 202–566–1077, (*e-mail: walker.lemuel@epa.gov*), or Meghan Hessenauer, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave., NW., Washington, DC 20460, 202–566–1040 (*e-mail: hessenauer.meghan@epa.gov*).

SUPPLEMENTARY INFORMATION:

A. General Information

1. Does this action apply to me?

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

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

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

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

1. *Submitting Confidential Business Information (CBI).* Do not submit this information to EPA through *http://www.regulations.gov* or e-mail. Clearly mark the part or all of the information that you claim to be CBI. For CBI information in a disk or CD–ROM that you mail to EPA, mark the outside of the disk or CD–ROM as CBI and then identify electronically within the disk or CD–ROM the specific information that is claimed as CBI. In addition to one complete version of the comment that includes information claimed as CBI, a copy of the comment that does not contain the information claimed as CBI must be submitted for inclusion in the public docket. Information so marked will not be disclosed except in accordance with procedures set forth in 40 CFR part 2.

2. *Tips for Preparing Your Comments.* When submitting comments, remember to:

- Identify the rulemaking by docket number and other identifying information (subject heading, **Federal Register** date and page number).
- Follow directions—The agency may ask you to respond to specific questions or organize comments by referencing a Code of Federal Regulations (CFR) part or section number.
- Explain why you agree or disagree, suggest alternatives, and substitute language for your requested changes.
- Describe any assumptions and provide any technical information and/or data that you used.
- If you estimate potential costs or burdens, explain how you arrived at your estimate in sufficient detail to allow for it to be reproduced.
- Provide specific examples to illustrate your concerns, and suggest alternatives.
- Explain your views as clearly as possible, avoiding the use of profanity or personal threats.
- Make sure to submit your comments by the comment period deadline identified.

C. Abbreviations and Acronyms Used in the Preamble and Proposed Rule Text

ASTM: ASTM International
ATP: Alternate Test Procedure
CFR: Code of Federal Regulations
CWA: Clean Water Act
EPA: Environmental Protection Agency
FLAA: Flame Atomic Absorption Spectroscopy
HRGC: High Resolution Gas Chromatography
HRMS: High Resolution Mass Spectrometry
ICP/AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP/MS: Inductively Coupled Plasma-Mass Spectrometry
MS: Mass Spectrometry
NPDES: National Pollutant Discharge Elimination System
QA: Quality Assurance
QC: Quality Control
SDWA: Safe Drinking Water Act
SM: Standard Methods
STGFAA: Stabilized Temperature Graphite Furnace Atomic Absorption Spectroscopy
USGS: United States Geological Survey
VCSB: Voluntary Consensus Standards Body
WET: Whole Effluent Toxicity

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IV. References

I. Statutory Authority

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

II. Summary of Proposed Rule

EPA's regulations at 40 CFR part 136 identify test procedures that must be used for the analysis of pollutants in all applications and report under the CWA NPDES program as well as State certifications pursuant to section 401 of the CWA. Included among the approved test procedures are analytical methods developed by EPA as well as methods developed by voluntary standards development organizations such as ASTM International and by the joint efforts of the Standard Methods Committee which is comprised of three technical societies (American Public Health Association, American Water Works Association and the Water Environment Federation) and produce *Standard Methods for the Examination of Water and Wastewater.* EPA approves analytical methods (test procedures) for measuring regulated pollutants in wastewater. Regulated and regulatory entities use these approved methods for determining compliance with NPDES permits or other monitoring requirements. Often, these entities have a choice in deciding which approved method they will use because EPA has approved the use of more than one method. This rule proposes to add to this list of approved methods. Associated with the proposed approved methods are their regulated analytes (parameters) within the method. Some of these proposed methods introduce new technologies to the NPDES program, while others are updated versions of previously approved methods. These additions will improve data quality and provide the regulated community with greater flexibility. Further, EPA is aware that organizations sometimes republish methods to correct errors or revise the description. These changes do not affect the performance of the method. Therefore, if there are changes for methods in this proposed rule before publication of a final rule, EPA will include the updated versions. In the tables at Section 136.3, EPA lists the parameters in alphabetical order. To better identify new parameters proposed in this rule EPA added some of these parameters, such as bisphenol A and nonylphenol, at the end of these lists. In the final rule, EPA may choose to reorder the listings to arrange all parameters alphabetically.

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

EPA is proposing to add new EPA methods that require new technologies to its Part 136 test procedures. EPA also is proposing new versions of already approved EPA methods with technologies that have been in use for many years. The new EPA methods and new versions of EPA approved methods are described in the following paragraphs.

1. EPA is proposing a new version of EPA Method 1664, 1664B: 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 for use in CWA programs. In addition, EPA is proposing to amend the RCRA regulations at 40 CFR 260.11, which currently specify use of method 1664A, to additionally specify the revised version, 1664B.

Currently, Method 1664A is used as a required testing method to determine eligibility of materials for certain conditional exclusions from RCRA regulations under 40 CFR 260.20 and 260.22. These exclusions are known as "delistings." These delistings provide that certain wastes generated at particular facilities are no longer classified as hazardous wastes under RCRA. When delistings are granted by EPA, the Agency describes them, along with applicable conditions, in appendix IX to 40 CFR part 261.

A number of delistings specify, among other things, the following test method: "Method 9070A (uses EPA Method 1664, Rev. A)." This testing method must be used by waste generators to determine if their wastes are an oily waste for delisting purposes. The language used in Appendix IX reads this way because Method 9070A in SW–846 (including on the SW–846 Web site, *http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/9070a.pdf*) simply reads that Method 1664A is to be used. Thus, although Method 9070A is cited, it is actually Method 1664A. Method 9070A does not exist independently of Method 1664A.

Once this rule becomes final, we would encourage future delistings, if applicable, to cite the test method as "Method 9070A (uses Method EPA 1664, Rev. B)." EPA is not proposing to amend delistings granted in previous years that reference Method 1664A at this time, since it would require additional review to assess the need for such a change and an analysis of each delisting.

Oil and Grease is a method-defined parameter that measures hexane extractable material (HEM) using n-hexane (85% minimum purity, 99.0% minimum saturated C6 isomer, residue < 1mg/L.) Before the use of Freon® was banned, EPA defined oil and grease as Freon®-extractable material. To replace Freon® for oil and grease determinations (64 FR 26315, May 14, 1999) EPA conducted extensive side-by-side studies of several extracting solvents on a variety of samples to determine how the values compared to Freon®-extractable material values.

In today's proposed rule, EPA describes six oil and grease methods, and proposes only the three methods in Table IB that use n-hexane to extract the sample because the solvent-defined definition of oil and grease measurements precludes use of any other extraction solvent or extraction technique. Without extensive side-by-side testing, permit writers, permitees, and data reviewers lack a basis for comparing HEM permit limits or measurements to values obtained with other extraction solvents or techniques. EPA lacks information about whether permit writers or permitees would value having more ways to extract oil and grease samples, or about how much effort they or others would be willing to exert to determine if the alternate values were equal to HEM values or convertible to HEM values by a conversion factor.

Although solvents may not be changed, EPA has described some allowable changes to the proposed EPA Method 1664B. This method describes (1) modifications allowable for nationwide use without prior EPA reviews (cf. documentation procedures described at 40 CFR 136.6), and (2) describes modifications not allowable including the use of any extraction solvent other than n-hexane or determination technique other than gravimetry. Although Method 1664B allows use of alternate extraction techniques, such as solid phase extraction (SPE) some discharges or waste streams may not be amenable to SPE. For these samples, 1664B should be applied as written. Conditioning of the solid-phase disk or device with solvents other than n-hexane (*e.g.,* alcohol, acetone, etc) is allowed, only if this solvent(s) is completely removed from the SPE disk or device prior to passing the sample through the SPE disk or device.

2. EPA is proposing to include in Table IB new EPA Method 200.5 and clarifying that the axial orientation of the torch is allowed for use with EPA Method 200.7. EPA Method 200.5 "Determination of Trace Elements in

Drinking Water by Axially Viewed Inductively Coupled Plasma—Atomic Emission Spectrometry" employs a plasma torch viewed in the axial orientation to measure chemical elements (metals). It also includes performance data for the axial configuration that is not in Method 200.7 because the axial technology torch results were not available when Method 200.7 was developed. For some elements the axial orientation results in greater sensitivity and lower detection limits than the radial orientation. EPA now authorizes the use of Method 200.5 in testing under its Safe Drinking Water Act Program (73 FR 31616, June 6, 2008). Approval of Method 200.5 and the flexibility within Method 200.7 will allow laboratories to use either axial instruments or radial instruments to measure metals in water samples.

3. EPA is proposing to add EPA Method 525.2, an updated version of EPA Method 525.1, in Table IG (Test Methods for Pesticide Active Ingredients) as an additional approved method for all parameters for which EPA has previously approved Method 525.1. Further, EPA is soliciting comment on whether EPA should substitute Method 525.2 for Method 525.1.

EPA is proposing to include Pesticide Methods from Table IG in Table ID (Test Procedures for Pesticides). Specifically, EPA is proposing to add EPA Method 525.2 for the same pesticides for which EPA has approved Method 525.1 in Table IG. Both methods use GC/MS methodology.

EPA is proposing to add some of the Pesticide Active Ingredients methods in Table IG that have been in use for more than 10 years to Table ID for general use. These methods are:

a. EPA Method 608.1, "The Determination of Organochloride Pesticides in Municipal and Industrial Wastewater." This is a gas chromatographic (GC) method used to determine certain organochlorine pesticide compounds listed in industrial and municipal discharges. This method measures chlorobenzilate, chloroneb, chloropropylate, dibromochloropropane, etridiazole, PCNB, and propachlor.

b. EPA Method 608.2, "The Determination of Certain Organochlorine Pesticides in Municipal and Industrial Wastewater." This is a GC method used to determine certain organochlorine pesticides compounds in industrial and municipal discharges. This method measures chlorothalonil, DCPA, dichloran, methoxychlor, and permethrin.

c. EPA Method 614, "The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater." This is a GC method used to determine organophosphorus compounds in industrial and municipal discharges. This method measures azinphos methyl, demeton, diazinon, disulfoton, ethion, malathion, parthion methyl, and parathion ethyl.

d. EPA Method 614.1, "The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater." This is a GC method used to determine organophosphorus compounds in industrial and municipal discharges. This method measures dioxathion, EPN, ethion, and terbufos.

e. EPA Method 615, "The Determination of Chlorinated Herbicides in Municipal and Industrial Wastewater." This is a GC method used to determine chlorinated herbicides compounds in industrial and municipal discharges. This method measures 2,4-D, dalapon, 2,4-DB, dicamba, dichlorprop, dinoseb, MCPA, MCPP, 2,4,5-T, and 2,4,5-TP.

f. EPA Method 617, "The Determination of Organohalide Pesticides and PCBs in Municipal and Industrial Wastewater." This is a GC method used to determine organohalide compounds in industrial and municipal discharges. This method measures aldrin, α-BHC, β-BHC, γ-BHC (lindane), captan, carbophenothion, chlordane, 4,4′-DDD, 4,4′-DDE, 4,4′-DDT, dichloran, dicofol, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide, isodrin, methoxychlor, mirex, PCNB, perthane, strobane, toxaphene, trifluralin, PCB–1016, PCB–1221, PCB–1232, PCB–1242, PCB–1248, PCB–1254, and PCB–1260.

g. EPA Method 619, "The Determination of Triazine Pesticides in Municipal and Industrial Wastewater." This is a GC method used to determine triazine pesticides compounds in industrial and municipal discharges. This method measures ametryn, atraton, atrazine, prometon, prometryn, propazine, sec-bumeton, simetryn, simazine, terbuthylazine, terbutryn.

h. EPA Method 622, "The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater." This is a GC method used to determine organophosphorus pesticides compounds in industrial and municipal discharges. This method measures azinphos methyl, bolstar, chlorpyrifos, chlorpyrifos methyl, coumaphos, demeton, diazinon, dichlorvos, disulfoton, ethoprop, fensulfothion, fenthion, merphos, mevinphos, naled, parathion methyl, phorate, ronnel, stirofos, tokuthion, and trichloronate.

i. EPA Method 622.1, "The Determination of Thiophosphate Pesticides in Municipal and Industrial Wastewater." This is a GC method used to determine thiophosphate pesticides compounds in municipal and industrial discharges. This method measures aspon, dichlofenthion, famphur, fenitrothion, fonophos, phosmet, and thionazin.

j. EPA Method 632, "The Determination of Carbamate and Urea Pesticides in Municipal and Industrial Wastewater." This is a high-performance liquid chromatographic (HPLC) method used to determine carbamate and urea pesticide compounds in industrial and municipal discharges. This method measures aminocarb, barban, carbaryl, carbofuran, chlorpropham, diuron, fenuron, fenuron-TCA, fluometuron, linuron, methiocarb, methomyl, mexacarbate, monuron, neburon, oxamyl, propham, propoxur, siduron, swep.

4. EPA is proposing to add in Table IC EPA Method 1614A, "Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by HRGC/HRMS." EPA developed this method to determine 49 polybrominated diphenyl ether (PBDE) congeners in aqueous, solid, tissue, and multi-phase matrices. These ethers are used in brominated flame retardants. This method uses isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). This method allows use of a temperature-programmed injector/vaporizer and a short column to improve recoveries of the octa-, nona-, and decabrominated diphenyl ethers.

5. EPA is proposing to add in Table IC EPA Method 1668C, "Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS." This method determines individual chlorinated biphenyl congeners in environmental samples by isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). Current Part 136 methods only measure a mixture of congeners in seven Aroclors—PCB–1016, PCB–1221, PCB–1232, PCB–1242, PCB–1248, PCB–1254, and PCB–1260. EPA Method 1668C can measure the 209 individual PCB congeners in these mixtures. EPA developed Method 1668 for use in wastewater, surface water, soil, sediment, biosolids, and tissue matrices.

EPA first published Method 1668 in 1999 and it is being used in several environmental applications, including

NPDES permits. EPA based today's proposed version, 1668C, on the results of an interlaboratory validation study (EPA 2010a, b), peer reviews (EPA 2010c), and user experiences. In the development and subsequent multi-laboratory validation of this method, EPA has evaluated method performance characteristics, such as selectivity, calibration, bias, precision, quantitation and detection limits. For example, EPA has observed that detection limits and quantitation levels are usually dependent on the level of interferences and laboratory background levels rather than instrumental limitations. Thus, the published minimum levels of quantitation are conservative estimates of the concentrations at which a congener can be measured with laboratory contamination present (EPA 2010d).

EPA recognizes that the performance of this Method may vary among the 209 congeners, and in different matrices. This is typical of multi-analyte methods because not all chemicals respond identically to extraction and clean up techniques, or have identical instrument responses. In a study of data comparability between two laboratories on samples collected from the Passaic River in New Jersey, in which 151 PCB congeners were identified and measured, accuracy as measured by analysis of a NIST SRM was 15% or better. Recoveries of the PCB congeners ranged from 90% to 124% and averaged 105%; precision ranged from 4.2% to 23% (Passaic River 2010).

This PCB method and the polybrominated diphenyl ether (PBDE) Method 1614A are performance-based methods. This means that users have the flexibility to modify the method to adapt to the sometimes unique characteristics of the user's sample. There is flexibility to modify the sample preparation steps to remove substances that interfere with measurement of the PCB congeners. A consequence of this flexibility is that, after customizing a performance-based method for a specific sample or application, the user should continue to use the same customized procedures on these samples or applications to maintain data comparability.

EPA Method 1668C, the interlaboratory study report, and peer reviews are in the docket for today's rule and on EPA's CWA methods Web site at *http://www.epa.gov/waterscience/methods.* EPA lists Method 1668C in Table IC as the parameter, "PCBs 209 Congeners."

6. EPA is proposing to update in Table IH EPA Method 1622, "Cryptosporidium in Water by Filtration/IMS/FA" and EPA Method 1623, "Cryptosporidium and Giardia in Water by Filtration/IMS/FA" to reflect changes made in the December 2005 versions of these methods. EPA's drinking water program uses the 2005 versions of the methods. The methods allow the flexibility to choose among several types of filters, quality controls, and stains, as well as clarification on measuring sample temperatures, quality control sample requirements and use of quality control sample results, minimizing carry-over debris, analyst verification procedures and sample condition criteria upon receipt. This method substitution necessitates a change in the holding temperature (Table II) for *Cryptosporidium and Giardia* from 0–8 °C to refrigerate between 1–10 °C.

7. EPA is proposing in Table IH revised versions of EPA Methods 1103.1, 1106.1, 1600 (also in Table IA), 1603, and 1680 to correct technical errors. Specifically, for Methods 1103.1 and 1603, tryptone broth should be tryptone water (section 12.4.3). In addition, in Tables 2 and 3, respectively, of these two methods, the positive control organism for the cytochrome oxidase reagent has been changed to *P. aeruginosa* from *E. faecalis,* and the negative control organism for Simmons citrate agar has been changed to *S. flexneri* from *E. coli* for more definitive results. In section 7.5.2 of Method 1603, the formula for magnesium chloride hexahydrate should have a dot before the waters rather than an alpha sign ($MgCl_2{\cdot}6H_2O$). In Methods 1106.1 and 1600, in Tables 6 and 7, respectively, the true spiked Enterococci "T (CFU/100 mL)" in the spiked sample based on the lot mean valued provided by the manufacturer should be 32 instead of 11.2. In Method 1680, the lactose for Lauryl Tryptose Broth (LTB) should be 5.0 g, not 25.0 g (section 7.6.1), and the dipotassium hydrogen phosphate for EC medium should be 4.0 g, not 44.0 g (section 7.7.1).

8. EPA is proposing to add Method 1627, "Kinetic Test Method for the Prediction of Mine Drainage Quality." The method is a standardized simulated weathering test that provides information to predict the quality of mine drainage from coal mining operations or weathering. The method also can be a tool with which to generate data in the design and implementation of best management practices and treatment processes needed by mining operations to meet U.S. EPA discharge requirements at 40 CFR part 434. Other publications have referred to this method generically as the ADTI Weathering Procedure 2 (ADTI–WP2). EPA lists Method 1627 in Table IB as "Acid Mine Drainage." The method is suitable for determinations of probable hydrologic consequences and to develop cumulative hydrologic impact assessment data to support Surface Mining Control and Reclamation Act (SMCRA) permit application requirements. Although this method is directed toward the coal mining industry and regulatory agencies, the method may be applicable to highway and other construction involving cut and fill of potentially acid-producing rock. This method may be used to predict the water quality characteristics (*e.g.,* pH, acidity, metals) of mine site discharges using observations from sample behavior under simulated and controlled weathering conditions. The method was developed and evaluated in single, multiple and interlaboratory method validation studies in laboratories representing the mining industry, private sector, federal agencies, and academia.

9. EPA proposes to approve EPA Method 624, "Purgeables," for definitive measurements of acrolein and acrylonitrile in wastewater. Currently this method is approved only to screen samples for the presence of acrolein and acrylonitrile. Footnote 4 to Table IC requires that the analyst confirm occurrences with either EPA Method 603 or 1624 because, when EPA promulgated this method, EPA believed the confirmatory step was necessary. Commenters on a previous proposed rule to amend part 136 (69 FR 18166, April 6, 2004) requested that EPA allow use of Method 624 for definitive determination of acrolein and acrylonitrile in wastewater without a confirmatory step and provided EPA with data. EPA has considered this comment and after reviewing additional data (Test America 1, 2) is proposing to revise the listing of Method 624 in Table IC to remove footnote 4 that requires a confirmatory analysis.

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

EPA is proposing to revise how we identify approved methods that are published by the Standard Methods Committee. Currently in the tables at 136.3(a), EPA lists these methods in one or more columns as being in the 18th, 19th, 20th printed compendiums, or in the On-line editions published by the Standard Methods Committee. EPA identifies which versions are approved by the printed edition in which the method is published or, in the case of the electronic version of the method, by the last two digits of the year in which the method was published by the Standard Methods Committee (*e.g.,* Standard Method 2320 B–97). In some cases, EPA has approved more than one version of a Standard Method. Approval of several versions of the same Standard Method has led to inconsistencies in how laboratories conduct these analyses especially in quality assurance/quality control (QA/QC) practices. For this reason, EPA is proposing to approve only the most recent version of a method published by the Standard Methods Committee with as few exceptions as possible by listing only one version of the method with the year of publication designated by the last four digits in the method number (*e.g.,* Standard Method 2320 B–1997). This change allows use of a specific method in any edition that includes a method with the same method number and year of publication. Previously, a laboratory only could use the method that was published in the edition of *Standard Methods* listed in the tables at 136.3(a). In some cases, EPA used footnotes to designate approved Standard Methods that are no longer published in *Standard Methods.*

In addition, EPA is proposing to approve new Standard Methods, SM, new versions of currently approved SM, and the use of an already approved SM for a chemical that is not currently listed in Table IB. The new versions of currently approved SM have been revised to clarify or improve the instructions in the method, improve the quality control (QC) instructions, or make editorial corrections. The proposed new SM and new versions of SM are described in the following paragraphs.

1. EPA is proposing to add SM 5520 B–2001 and SM 5520 F–2001 for Oil and Grease determinations. These methods measure hexane extractable material (HEM). EPA is proposing these methods because they use n-hexane as the extraction solvent. EPA is not proposing SM 5520 G–2001 because it allows use of a co-solvent, such as acetone. In the preceding description of EPA's proposed Method 1664B, EPA explained that oil and grease is a measurement defined by the solvent, in this case n-hexane, used to extract oil and grease from the sample. Thus, use of any other solvent system, such as a co-solvent is precluded.

2. EPA is proposing to add SM 4500–NH_3 G–1997, Ammonia (as N) and TKN, Phenate Method, which is an automated version of the previous version of a previously approved SM 4500–NH_3 F–1997.

3. EPA is proposing to add SM 4500–B B–2000, Boron, Curcumin Method, which uses the same chemistry and instruments as Method I–3112–85.

4. EPA is proposing to add SM 4140–1997, Inorganic Ions (Bromide, Chloride, Fluoride, Orthophosphate, and Sulfate), Capillary Ion Electrophoresis with Indirect UV Detection, which uses the same technology as the EPA approved ASTM Method D6508–00.

5. EPA is proposing to add SM 3114 C–2009, Arsenic and Selenium by Continuous Hydride Generation/Atomic Absorption Spectrometry, which is an automated version of the approved manual method, and uses the same technology as Method I–2062–85.

6. EPA is proposing to add SM 3111 E–1999 for determinations of aluminum and beryllium. The method uses the same instrumental techniques as SM 3111D with an additional chelation concentration step for increased sensitivity.

7. EPA is proposing to add SM 5220 B–1997 for Chemical Oxygen Demand which is similar to EPA Method 410.3.

8. EPA is proposing to add SM 4500 N_{ORG} D–1997 for determinations of Kjeldahl Nitrogen—Total, which has a similar chemical and instrument setup as in EPA Method 351.2 in Table IB. The same chemical reaction is measured in both of these methods.

9. EPA is proposing to add SM 4500 P G–1999 and SM 4500 P H–1999, Phosphorus. Both of these methods use separate flow injection instrumentation that is the same as EPA Method 365.1.

10. EPA is proposing to add SM 4500 P E–1999 and SM 4500 P F–1999, Phosphorus. These methods, 4500 P E–1999 Manual Single Reagent and F–1999 Automated Ascorbic Acid, have been approved for drinking water analyses (73 FR 31616, June 3, 2008).

11. EPA is proposing to add SM 4500 O B, D, E and F–2001, Oxygen, Iodometric Methods. EPA is proposing these methods because Standard Methods has broken down the Winkler titration method into several sections. Sections 4500 O B, D, E and F have been added to provide a more detailed Winkler titration. Section B contains information on how to collect the sample and what pretreatment may be needed for just the Winkler titrations. Sections D, E, and F contain specific sample pretreatment for interferences. Section D (see Item 12) is for ferrous iron interferences. Section E (*see* Item 13) is for samples with a high concentration of Total Suspended Solids. Section F is for samples with large concentrations of biological solids. These sections are similar to the instructions in ASTM D888, AOAC 973.45, and USGS I–1575–78.

12. EPA is proposing to add SM 4500 O D–2001, Oxygen, Permanganate Modification. This method for determinations of dissolved oxygen contains the same permanganate pretreatment step that is specified in ASTM D 888 and AOAC 973.45.

13. EPA is proposing to add SM 4500 O E–2001, Oxygen, Alum Flocculation Modification. This method for dissolved oxygen describes a pretreatment step that removes high concentrations of suspended solids.

14. EPA is proposing to add SM 3500 K C–1997, Potassium, Selective Electrode Method. This method uses the same electrochemical procedure to measure Potassium that is used in the Standard Methods for ammonia, chloride, cyanide, and nitrate. Only the electrode construction is different.

15. EPA is proposing to add SM 2540 E–1997 for determinations of Residues—Volatile. This fixed and volatile solids method uses the same equipment and procedures to measure this method defined parameter as approved EPA Method 160.4.

16. EPA is proposing to add SM 4500 SiO_2 E–1997 and SM 4500 SiO_2 F–1997, Silica. These methods have the same instrument setup and molybdate color reagent as USGS Method I–2700, but utilize different reducing agents to produce molybdenum blue color. There are slight modifications in the chemical reaction, but the molybdenum blue final analyte is the same.

17. EPA is proposing to add SM 4500 SO_4 C–1997, D–1997, E–1997, F–1997 and G–1997, Sulfate. EPA is proposing to approve the online version of these methods because they are identical to the approved versions published in the 18th, 19th and 20th edition of Standard Methods. EPA approved the online versions for drinking water use (73 FR 31616, June 3, 2008).

18. EPA is proposing to add SM 4500 S^{2-} B–2000 and C–2000, Sulfide. These approved methods have been revised to describe more completely the sample collection, transportation and analysis steps.

C. Changes to 40 CFR 136.3 To Include New ASTM Methods and New Versions of Previously Approved ASTM Methods

EPA is proposing to add to the list of approved testing procedures new ASTM methods for existing pollutants in Table IB, such as cyanide, and methods for new pollutants, such as the nonylphenols in Table IC. EPA also is proposing new versions of previously approved ASTM methods.

1. EPA is proposing to add ASTM D2036–09 Standard Test Methods for Cyanides in Water, Test Method A Total Cyanide after Distillation. In 2009, ASTM revised the version of this method currently listed in part 136. The method measures cyanides that are free, and strong-metal-cyanide complexes (*e.g.* iron cyanides) that dissociate and release free cyanide when refluxed under strongly acidic conditions. The cyanide in some cyano complexes of transition metals, for example, cobalt, gold, platinum, etc., is not determined. Samples are digested with sulfuric acid in the presence of magnesium chloride in a distillation reaction vessel that consists of a 1–L round bottom flask, with provision for an inlet tube and a condenser connected to a vacuum-type absorber. The flask is heated with an electric heater. Smaller distillation tubes such as 50-mL midi tubes or 6-mL MicroDist™ tubes described in D7284–08 can be used if the quality control requirements in D2036–09 are satisfied. After distillation, the cyanide concentration can be determined with titration, ion chromatography, colorimetric procedure (spectrophotometric), selective ion electrode, or flow injection analysis with gas diffusion separation and amperometric detection. The inclusion of ion chromatography and gas diffusion separation with amperometric detection as determinative steps (D2036–09, sections 16.5 and 16.6) will give users additional options to measure cyanide after distillation. Furthermore, these determinative steps can be used to mitigate interferences that have been associated with conventional colorimetric test methods.

2. EPA is proposing to add ASTM D6888–09 Standard Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection. This method is used to determine the concentration of available inorganic cyanide in an aqueous wastewater or effluent. The method detects the cyanides that are free and metal-cyanide complexes that are easily dissociated into free cyanide ions. The method does not detect the less toxic strong metal-cyanide complexes, cyanides that are not "amenable to chlorination." Total cyanide can be determined for samples that have been distilled as described in Test Methods D2036–09, Test Method A, Total Cyanides after Distillation. Complex cyanides bound with nickel or mercury are released by ligand displacement with the addition of a ligand displacement agent prior to analysis. Other available cyanide species do not require ligand displacement under the test conditions. If samples are distilled for total cyanide, ligand exchange reagents are not required since the cyanide complexes are dissociated and absorbed into the sodium hydroxide capture solution during distillation. The treated or distilled sample is introduced into a flow injection analysis (FIA) system where it is acidified to form hydrogen cyanide. The hydrogen cyanide gas diffuses through a hydrophobic gas diffusion membrane, from the acidic donor stream into an alkaline acceptor stream. Up to 50-mg/L sulfide is removed during flow injection to mitigate sulfide interference. The captured cyanide is sent to an amperometric flow cell detector with a silver-working electrode. In the presence of cyanide, silver in the working electrode is oxidized at the applied potential. The anodic current measured is proportional to the concentration of cyanide in the standard or sample injected.

3. EPA is proposing to add ASTM D7284–08 Standard Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection. This method determines the concentration of total cyanide in wastewater, and detects the cyanides that are free and strong-metal-cyanide complexes (*e.g.,* iron cyanides) that dissociate and release free cyanide when refluxed under strongly acidic conditions. This method has a range of approximately 2 to 400 μg/L (parts per billion) total cyanide. Higher concentrations can be measured with sample dilution or lower injection volume. The determinative step of this method utilizes flow injection with amperometric detection based on ASTM D6888–09. Sample distillation is based on Lachat QuikChem Method 10–204–00–1–X. Prior to analysis, samples must be distilled with a micro-distillation apparatus described in the test method or with a suitable cyanide distillation apparatus specified in Test Methods D 2036–09. The samples are distilled with a strong acid in the presence of magnesium chloride catalyst and captured in sodium hydroxide absorber solution. The absorber solution from the distillation is introduced into a flow injection analysis (FIA) system where it is acidified to form hydrogen cyanide. The hydrogen cyanide gas diffuses through a hydrophobic gas diffusion membrane, from the acidic donor stream into an alkaline acceptor stream. The captured cyanide is sent to an amperometric flow cell detector with a silver-working electrode. In the presence of cyanide, silver in the working electrode is oxidized at the applied potential. The anodic current measured is proportional to the concentration of cyanide. This method has been shown to be less susceptible to interferences compared to conventional spectrophotometric determinations for total cyanide.

4. EPA is proposing to add ASTM D7511–09e2 Standard Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection. This method determines the concentration of total cyanide in drinking and surface waters, as well as domestic and industrial wastes. Cyanide ion (CN-), hydrogen cyanide in water (HCN(aq)), and the cyano-complexes of zinc, copper, cadmium, mercury, nickel, silver, and iron may be determined by this method. Cyanide ions from Au(I), Co(III), Pd(II), and Ru(II) complexes are only partially determined. The applicable range of the method is 3 to 500 μg/L cyanide using a 200-μL sample loop. The range can be extended to analyze higher concentrations by sample dilution or by changing the sample loop volume. ASTM D7511–09e2 decomposes complex cyanides by narrow band, low watt UV irradiation in a continuously flowing acidic stream at room temperature. Reducing and complexing reagents, combined with the room temperature narrow band low watt UV, minimize interferences. The hydrogen cyanide generated passes through a hydrophobic membrane into a basic carrier stream. The cyanide concentration is determined by amperometry. This method operates similarly to available cyanide methods OIA1677 and ASTM D6888–09. The available cyanide methods employ a preliminary ligand addition to liberate cyanide ion from weak to moderate metal cyanide complexes. These available cyanide methods were developed because they overcome significant interferences caused by the preliminary chlorination and/or distillation processes. Instead of ligands, ASTM D7511–09e2 irradiates the sample causing strong metal cyanide complexes plus all complexes measured by the available cyanide methods to liberate cyanide and generate hydrogen cyanide. Once the sample solution passes from the UV irradiation, the measurement principle is equivalent to OIA1677 and/or ASTM D6888–09.

5. Because there were no EPA-approved methods for free cyanide when water quality criteria were

established for free cyanide EPA recommended measurement of cyanide after a "total" distillation. Analytical methods for free cyanide have been developed, and in today's rule EPA is proposing to add free cyanide as a parameter (24A in Table IB.) For determinations of this parameter, EPA is proposing to allow use of the approved available cyanide method, OIA 1677–09, and two ASTM methods (D4282–02 and D7237–10.) ASTM D4282–02 Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion determines free cyanide as the cyanide that diffuses into a sodium hydroxide solution from a solution at pH 6. It is not applicable to cyanide complexes that resist dissociation, such as hexacyanoferrates and gold cyanide, and it does not include thiocyanate and cyanohydrin. ASTM D7237–10 Standard Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection determines free cyanide with the same instrumentation and technology as approved methods, ASTM D6888–09 and OIA 1677–09, but under milder (less acidic) conditions and without use of ligand replacement reagents.

6. EPA is proposing to add ASTM D888–09 Standard Test Method for Dissolved Oxygen in Water. This method determines dissolved oxygen concentrations in water using the titrimetric (Part A), polarographic (Part B) and luminescence-based (Part C) detection methods. This standard test method is applicable to the determination of dissolved oxygen between 0.05–20 ppm in influent, effluent or ambient water testing. ASTM recently updated Part C of this method to include a detailed description of the technology and to update calibration procedures to include a two-point calibration and an air saturated water calibration in addition to a water saturated air calibration. This method may be used for Biological Oxygen Demand (BOD) and Carbonaceous Oxygen Demand (CBOD.)

7. EPA is proposing to add ASTM D7573–09 Standard Test Method for Total Carbon and Organic Carbon in Water by High Temperature Catalytic Combustion and Infrared Detection. This Method has the same chemical and instrument setup as approved SM 5310 B–2000.

8. EPA is proposing to add in Table IC ASTM D7065–06: Standard Test Method for Determination of five chemicals: Nonylphenol (NP), Bisphenol A (BPA), p-tert-Octylphenol (OP), Nonylphenol Monoethoxylate (NP1EO), and Nonylphenol Diethoxylate (NP2EO) in Environmental Waters by Gas Chromatography Mass Spectrometry. These five chemicals are partitioned into an organic solvent, separated using gas chromatography and detected with mass selective detection. These chemicals or isomer mixtures are qualitatively and quantitatively determined. Although this method adheres to selected ion monitoring mass spectrometry, full scan mass spectrometry has also been shown to work well under these conditions. This method has been multi-laboratory validated for use with surface water and waste treatment effluent samples and is applicable to these matrices. It has not been investigated for use with salt water or solid sample matrices. The reporting limit for nonylphenol is 5 μg/L (ppb); the chronic Freshwater Aquatic Life Ambient Water Quality Criterion is 6.6 ppb.

9. EPA is proposing to add in Table IC ASTM D7574–09: Standard Test Method for Determination of BPA in Environmental Waters by Liquid Chromatography/Tandem Mass Spectrometry. BPA is an organic chemical produced in large quantities. BPA is soluble in water and undergoes degradation in the environment. The reporting limit for BPA is 20 ng/L which is fifty times less than the limit in D7065–06 (see preceding Item 8). The method is based on a solid phase extraction (SPE) followed by separation with liquid chromatography and tandem mass spectrometry (LC/MS/MS), which reduces the amount of sample required, solvents, the analysis time, and the reporting limits. The method has been tested in effluents from secondary and tertiary publicly owned treatment works (POTW), and fresh surface and ground water.

10. EPA is proposing to add in Table IC ASTM D7485–09: Standard Test Method for Determination of NP, OP, NP1EO, and NP2EO in Environmental Waters by Liquid Chromatography/Tandem Mass Spectrometry. The method extracts these four chemicals from water with SPE followed by LC/MS/MS separation and detection. These chemicals are qualitatively and quantitatively determined by this method. This method uses single reaction monitoring (SRM) mass spectrometry. Environmental waters tested using this method were sewage treatment plant effluent, river water, seawater, and a modified ASTM D5905 artificial wastewater. The reporting limit for nonylphenol is 100 ng/L, ppt. The Freshwater and Saltwater Aquatic Life Ambient acute criterion is 7.0 ppb, and the chronic criterion is 1.7 ppb.

11. EPA is not proposing to include in Table IB two ASTM oil and grease methods, D7066–04 and D7575–10 because neither method uses n-hexane to determine oil and grease as hexane extractable material (HEM). As previously explained in the discussion of Method 1664B, HEM is a measurement defined by the solvent (n-hexane) used to extract oil and grease from the sample. D7066–04 employs a proprietary solvent, S–316, a dimer/trimer of chlorotrifluoroethylene to measure S–316-extractable substances from an acidified sample. Method D7066 may be useful for determinations of total petroleum hydrocarbons (TPH). Although TPH has been measured in some applications, EPA has never included it as a Part 136 pollutant nor received any convincing evidence that it should do so. Although S–316 is not the same solvent as the fluorocarbon, Freon®, it is a fluorochlorohydrocarbon.

Instead of n-hexane, ASTM D7575–10 uses a different extracting process, an extracting membrane, followed by infrared measurement of the materials in the sample that can pass through the membrane. Several other steps in D7575–10 significantly differ from 1664 including: Use of 10-mL sample aliquot from sample bottle vs. entire contents of 1–L sample; homogenization of samples; and the challenge of pushing solid oil and grease samples through a membrane. The results of a multi-laboratory study (OSS 2009) that the developer conducted as part of ASTM's evaluation of D7575 are in the docket.

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

To promote method innovation, EPA maintains a program whereby method developers may apply for an EPA review and potentially for approval of alternate test procedures. This Alternate Test Procedure (ATP) program is described for Clean Water Act applications at Parts 136.4 and 136.5. EPA has reviewed and is proposing for nationwide use eight alternate test procedures. These proposed new methods include: Hach Company's Method 10360 Luminescence Measurement of Dissolved Oxygen (LDO®) in Water, In-Situ Incorporated's Method 1002–8–2009 Dissolved Oxygen (DO) Measurement by Optical Probe, Method 1003–8–2009 Biochemical Oxygen Demand (BOD) Measurement by Optical Probe, and Method 1004–8–2009 Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe August 2009, Mitchell Method M5271 and M5331 for measuring turbidity in wastewater; Thermo Scientific's Orion Method

AQ4500 for measuring turbidity in wastewater; and Systea Scientific, LLC's Systea Easy (1-Reagent) Nitrate Method. Descriptions of these new methods included for approval are as follows:

1. EPA is proposing to approve Hach Company's Method 10360 Luminescence Measurement of Dissolved Oxygen (LDO®) in wastewater, Revision 1.1 dated January 4, 2006. EPA has reviewed this method and the data generated in a multi-laboratory validation study performed by Hach Company and is proposing to approve it for use in measuring dissolved oxygen. EPA is also proposing to approve the Hach method 10360 to be used for Dissolved Oxygen (DO) when determining BOD and CBOD.

This method uses an optical probe to measure the light emission characteristics from a luminescence-based reaction that takes place at the sensor-water interface. A light emitting diode (LED) provides incident light required to excite the luminophore substrate. In the presence of dissolved oxygen, the reaction is suppressed. The resulting dynamic lifetime of the excited luminophore is evaluated and equated to DO concentration.

The method involves the following steps:

- Calibration of the probe using water-saturated air, and
- Measurement of the dissolved oxygen in the sample using the probe.

Approved methods for measuring dissolved oxygen are listed at 40 CFR 136.3, Table IB. The performance characteristics of the Hach Company Method 10360 were compared to the characteristics of the methods listed at 40 CFR 136.3, Table IB for measurement of dissolved oxygen. Because the Hach Company Method 10360 is equally effective relative to the methods already promulgated in the regulations, EPA is proposing to include this method in the list of methods approved for measuring dissolved oxygen concentrations in wastewater when determining BOD and CBOD.

2. EPA is proposing to approve In-Situ Incorporated's Method 1002–8–2009 Dissolved Oxygen Measurement by Optical Probe. EPA has reviewed this method and the data generated in a multi-laboratory validation study performed by In-Situ Incorporated and is proposing to approve it for use in measuring dissolved oxygen. In-Situ Method 1002–8–2009 uses a new form of electrode based on the luminescence emission of a photoactive chemical compound and the quenching of that emission by oxygen to measure dissolved oxygen concentration.

The method involves the following steps:

- Calibration of the probe using water-saturated air, and
- Measurement of the dissolved oxygen in the sample using the probe.

Approved methods for measuring dissolved oxygen are listed at 40 CFR 136.3, Table IB. The performance characteristics of the In Situ Method 1002–8–2009 were compared to the characteristics of the methods listed at 40 CFR 136.3, Table IB for measurement of dissolved oxygen. Because the In-Situ Method 1002–8–2009 is equally effective relative to the methods already promulgated in the regulations, EPA is proposing In-Situ Method 1002–8–2009 for inclusion in the list of methods approved for measuring dissolved oxygen concentrations in wastewater.

3. EPA is proposing to approve In-Situ Incorporated's Method 1003–8–2009 Biochemical Demand (BOD) Measurement by Optical Probe. EPA has reviewed this method and the data generated in a multi-laboratory validation study performed by In-Situ Incorporated and is proposing to approve it for measuring BOD.

In-Situ Method 1003–8–2009 uses a new form of electrode based on the luminescence emission of a photoactive chemical compound and the quenching of that emission by oxygen to measure dissolved oxygen concentration when performing the 5-day BOD test.

The method involves the following steps:

- Filling a BOD bottle with diluted seeded sample,
- Measuring the dissolved oxygen in the sample using an optical DO probe,
- Sealing and incubating the bottle for five days,
- Measuring the dissolved oxygen with an optical probe after the five day incubation period, and
- Calculating the BOD from the difference between the initial and final dissolved oxygen measurements.

Approved methods for measuring BOD are listed at 40 CFR 136.3, Table IB. The performance characteristics of In-Situ Method 1003–8–2009 were compared to the characteristics of the methods listed at 40 CFR 136.3, Table IB for measurement of BOD. Because In-Situ Method 1003–8–2009 is equally effective relative to the methods already promulgated in the regulations, EPA is proposing In-Situ Method 1003–8–2009 for inclusion in the list of methods approved for measuring BOD.

4. EPA is proposing to approve In-Situ Incorporated's Method 1004–8–2009 Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe. EPA has reviewed this method and the data generated in a multi-laboratory validation study performed by In-Situ Incorporated and is proposing to approve it for use in measuring carbonaceous biochemical oxygen demand (CBOD). In-Situ Method 1004–8–2009 uses a new form of electrode based on the luminescence emission of a photoactive chemical compound and the quenching of that emission by oxygen to measure dissolved oxygen concentration when performing the CBOD test.

The method involves the following steps:

- Filling a BOD bottle with diluted seeded sample,
- Adding a chemical nitrification inhibitor,
- Measuring the dissolved oxygen in the sample using an optical dissolved oxygen probe,
- Sealing and incubating the bottle for five days,
- Measuring the dissolved oxygen with an optical probe after the five day incubation period, and
- Calculating the CBOD from the difference between the initial and final dissolved oxygen measurements.

Approved methods for measuring CBOD are listed at 40 CFR 136.3, Table IB. The performance characteristics of In Situ-Method 1004–8–2009 were compared to the characteristics of the methods listed for measurement of CBOD. Because In-Situ Method 1004–8–2009 is equally effective relative to the methods already promulgated in the regulations, EPA is proposing In-Situ Method 1004–8–2009 for inclusion in the list of methods approved for measuring CBOD.

5. EPA is proposing to approve the Mitchell Method M5271 dated July 31, 2008. This method uses laser based nephelometry to measure turbidity in drinking water and wastewater. The method involves the following steps for instruments other than on-line continuous models:

- Mixing the sample to thoroughly disperse the solids,
- Waiting until air bubbles disappear,
- Pouring a sample into a turbidimeter tube, and
- Reading turbidity directly from the instrument scale or from the appropriate calibration curve.

Approved methods for turbidity are listed at 40 CFR 136.3 Table 1B. The performance characteristics of Mitchell Method M5271 were compared to the performance characteristics of EPA Method 180.1 listed at 40 CFR 136.3 for measurement of turbidity. Comparisons were based on results obtained from turbidimeters placed in series which took measurements at one minute intervals over a 20 to 30 hour time period at three different public water supply systems (in one case measurements were taken at 15 minute intervals). Testing included source water from one ground water source and two surface water sources and included at least one natural filter event (back-flush) in lieu of artificially calibrated spikes using a primary standard spiking solution. Additionally, a demonstration of performance at higher turbidities was conducted by making replicate measurements of primary standards at four levels (5 NTU, 10 NTU, 20 NTU and 40 NTU). Results showed excellent correlation between measurements made using a tungsten filament incandescent bulb as specified in EPA Method 180.1 and those made using the laser light source specified in Mitchell Method M5271. Based on the results of these studies, EPA has determined that Mitchell Method M5271 is as effective as the methods already promulgated in the regulations. EPA is proposing to add this method to the list of methods approved for measurement of turbidity in wastewater.

6. EPA is proposing Mitchell Method M5331 dated July 31, 2008. This method uses LED based nephelometry to measure turbidity. The method involves the following steps for instruments other than on-line continuous models:

- Mixing the sample to thoroughly disperse the solids,
- Waiting until air bubbles disappear,
- Pouring the sample into turbidimeter tube, and
- Reading turbidity directly from the instrument scale or from the appropriate calibration curve.

Approved methods for turbidity are listed at 40 CFR 136.1 Table 1B. The performance characteristics of Mitchell Method 5331 were compared to the performance characteristics of EPA Method 180.1 listed at 40 CFR 136.3 for measurement of turbidity. Comparisons were based on results obtained from turbidimeters placed in series, which took measurements at one minute intervals over a 20 to 30 hour time period at three different public water supply systems (in one case measurements were taken at 15 minute intervals). Testing included source water from one ground water source and two surface water sources and included at least one natural filter event (back-flush) in lieu of artificially calibrated spikes using a primary standard spiking solution. Additionally, a demonstration of performance at higher turbidities was conducted by making replicate measurements of primary standards at four levels (5 NTU, 10 NTU, 20 NTU and 40 NTU). Results showed excellent correlation between measurements made using a tungsten filament incandescent bulb as specified in EPA Method 180.1 and the LED light source specified in Mitchell Method M5331. Based on the results of these studies, EPA has determined that Mitchell Method M5331 is equally effective relative to the methods already promulgated in the regulations. EPA is proposing to add this method to the list of methods approved for measurement of turbidity in wastewater.

7. EPA is proposing to approve Thermo Scientific's Orion Method AQ4500 dated March 12, 2009. This method uses LED-based nephelometry to measure turbidity. The method involves the following steps:

- Calibration of the instrument using a primary calibration standard,
- Placing the sample into the sample chamber, and
- Reading the turbidity result displayed on the instrument.

Approved methods for turbidity are listed at 40 CFR 136.3 Table IB. The performance characteristics of Thermo Scientific's Orion Method AQ4500 were compared to the performance characteristics of EPA Method 180.1 listed at 40 CFR 136.3 for measurement of turbidity. Comparisons were based on an ASTM round robin study comparing results from analyses of 28 different samples of various types including formazin standards, styrene divinyl benzene (SDVB) co-polymer bead standards and real world samples ranging from approximately 2 NTU to over 1,000 NTU. These analyses were conducted using turbidimeters with various light sources including tungsten filament incandescent bulbs as specified in EPA Method 180.1 and white LEDs as specified in Thermo Scientific's Orion Method AQ4500. Additionally, a demonstration of performance at lower turbidities was conducted by making 20 replicate measurements of dilute formazin standards at four levels (0.2 NTU, 0.5 NTU, 1 NTU, and 2 NTU) using turbidimeters with tungsten filament incandescent bulbs as specified in EPA Method 180.1 and turbidimeters using white LEDs as specified in Thermo Scientific Orion Method AQ4500. Results showed significant correlation between measurements made using a tungsten filament incandescent bulb as specified in EPA Method 180.1 and those made using the LED light source specified in Thermo Scientific's Orion Method AQ4500. Based on the results of these studies, EPA has determined that Thermo Scientific's Orion Method AQ4500 is as effective as the methods already promulgated in the regulations. EPA is proposing to add this method to the list of methods approved for measurement of turbidity in wastewater.

8. EPA is proposing to approve Systea Scientific, LLC's Systea Easy (1-Reagent) Nitrate Method dated February 4, 2009. This is a method that uses automated discrete analysis, and spectrophotometry to determine concentrations of nitrate and nitrite combined or singly. The method involves the following steps:

- Reduction of nitrate in a sample to nitrite using a non-hazardous proprietary reagent,
- Diazotizing the nitrite originally in the sample plus the reduced nitrate with sulfanilamide followed by coupling with N-(1-napthyl) ethylenediamine dihydrochloride under acidic conditions to form a highly colored azo dye,
- Colorimetric determination in which the absorbance of color at 546 nm is directly proportional to the concentration of the nitrite plus the reduced nitrate in the sample,
- Measurement of nitrite singly, if needed, by analysis of the sample while eliminating the reduction step, and
- Subtraction of the nitrite value from that of the combined nitrate plus nitrite value to measure nitrate singly if needed.

Approved methods for nitrate, nitrite and combined nitrate/nitrite are listed at 40 CFR 136.3, Table 1B. The performance characteristics of the Systea Easy (1-Reagent) Nitrate Method were compared to the characteristics of the methods listed at 40 CFR 136.3 for nitrate and nitrite. Based on the results of the comparative studies, EPA has determined that the Systea Easy (1-Reagent) Nitrate Method is as effective as the methods already promulgated in the regulations for use in determining concentrations of nitrate and nitrite and combined nitrate/nitrite. The method is a "green" alternative to other approved methods that use cadmium, a known carcinogen, for the reduction of nitrate to nitrite. The performance of Systea Easy (1-Reagent) Nitrate Method is equivalent to other methods already approved for measurement of nitrate, nitrite and combined nitrate/nitrite in wastewater.

E. Clarifications and Corrections to Previously Approved Methods in 40 CFR 136.3

EPA is proposing a clarification to procedures for measuring orthophosphate, and is proposing to correct typographical or other citation errors in part 136.

1. EPA is clarifying the purpose of the immediate filtration requirement in

orthophosphate measurements, which is to assess the dissolved or bio-available form of orthophosphorus (*i.e.,* that which passes through a 0.45 micron filter), hence the requirement to filter the sample immediately upon collection. This filtration excludes any particulate forms of phosphorus that might hydrolyze into orthophosphorus in a slightly acidic sample during the allowed 48 hour holding time. Each grab sample must be filtered within 15 minutes of collection to prevent orthophosphate formation. Specifically, filtration may not be delayed until the final grab sample is collected; each grab sample must be filtered upon collection. However, the filtered grab samples may be held for compositing up to the 48-hour holding time.

2. EPA is proposing to correct missing citations to the table of microbiological methods for ambient water monitoring which are specified in Table IH at 40 CFR 136.3. Stakeholders asked EPA to separately specify the microbiological methods that EPA has approved for wastewater (Table IA) from those for ambient water. On August 15, 2005 (70 FR 48256), EPA proposed to move microbial (bacterial and protozoan) methods which were applicable to ambient water to a new table, Table IH. However, in the final rule of March 26, 2007 (72 FR 14220), EPA inadvertently omitted fecal coliform, total coliform, and fecal streptococcus methods from the table. EPA is proposing to add these methods to Table IH.

3. EPA is proposing to correct several other typographical or minor citation errors, such as incomplete or incorrect method citations.

F. Proposed Revisions in Table II at 40 CFR 136.3(e) to Required Containers, Preservation Techniques, and Holding Times

EPA is proposing revisions to Table II at 136.3(e) to clarify how to resolve conflicts between instructions in this table and instructions in an approved method or other source, and to amend some of the current requirements in Table II.

1. The introductory text to Table II at 136.3(e) specifies that the instructions in the table take precedence over other sources of this information. EPA publishes holding time and related instructions in Table II to provide a consistent set of instructions, and for other reasons. Not all methods contain complete instructions, and some otherwise equivalent methods (or methods for the same parameter) have conflicting instructions. For example, Table II instructions specify the 48 hour BOD holding time while some Part 136 methods recommend 24 hours. In this instance Table II instructions take precedence. EPA recognizes that there may be cases where new technologies or advancements in current technologies may produce approved methods with instructions for a specific parameter that differ from Table II instructions, and provide better results. Cyanide determinations and some automated methods may fall into this category. Therefore, EPA is proposing to revise the text at 136.3(e) to allow a party to submit documentation to their permitting or other authority that supports use of an alternative approach. EPA is proposing to revise the introductory text to the table to read as follows: "Information in this table takes precedence over instructions provided in specific methods or elsewhere unless a party documents the acceptability of an alternative to the Table II instructions. The nature, timing and extent of the required documentation (*i.e.* how to apply and review as well as the amount of supporting data) are left to the discretion of the permitting authority (State Agency or EPA Region) or other authority and may rely on instructions, such as those provided for method modifications at 136.6." Thus, an alternate sample container, preservation and/or holding time may be considered at the discretion of the permitting authority or other authority.

2. Some stakeholders have asked EPA to extend the holding time for *Escherichia coli* and *Enterococcus.* In 2006, EPA conducted a nationwide holding time study (EPA 2006) for fresh and marine ambient waters and concluded that, on a nationwide basis, the Agency was unable to justify extending the holding time for *Escherichia coli* or *Enterococcus* in these water matrices. However, EPA is proposing to provide some relief by revising footnote 22 to Table II, which applies to bacterial tests. This footnote currently reads as follows: "Sample analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed (in incubator) within 2 hours of receipt at the laboratory."

Stakeholders have commented that laboratories must meet the two-hour analysis start time, even if they receive the samples early enough that they could start after two hours and still meet the overall six-hour time limit. EPA is proposing to revise the footnote to read "Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection."

3. EPA is proposing to revise the cyanide sample handling instructions in Footnote 5 of Table II to recommend the treatment options for samples containing oxidants described in ASTM's sample handling practice for cyanide samples, D7365–09a. This practice advises analysts to add a reducing agent only if an oxidant is present, and use of the reducing agents sodium thiosulfate ($Na_2S_2O_3$), ascorbic acid, sodium arsenite ($NaAsO_2$), or sodium borohydride ($NaBH_4$).

4. EPA is proposing to revise the cyanide sample handling instructions in Footnote 6 of Table II to describe options available when the interference mitigation instructions in D7365–09a are not effective. EPA proposes to allow use of any technique for removal or suppression of interference, provided the laboratory demonstrates and documents that the alternate technique more accurately measures cyanide through quality control measures described in the analytical test method.

5. EPA is proposing to revise footnote 16 of Table II instructions for handling Whole Effluent Toxicity (WET) samples to be consistent with the November 19, 2002 (67 FR 69951) "Guidelines for Establishing Test Procedures for the Analysis of Pollutants; Whole Effluent Toxicity Test Methods; Final Rule," as well as the three toxicity methods (Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (5th Edition, October 2002), Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (4th Edition, October 2002), and Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms (3rd Edition, October 2002). In the 2002 final rule, EPA established the acceptable range for the current sampling holding temperature for aquatic toxicity tests as 0 to 6 °C based on current National Environmental Laboratory Accreditation Conference (NELAC) standards. EPA also clarified in the final rule that hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation. Section 8.5.1 of all three WET methods listed previously states, "Unless the samples are used in an on-site toxicity test the day of collection (or hand delivered to the testing laboratory for use on the day of collection) it is recommended that they be held at 0 to 6 °C until used to inhibit microbial degradation, chemical transformation, and loss of highly volatile toxic substances." EPA is proposing to add two sentences to the

end of Footnote 16 of Table II based on this information. The two sentences are "Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation." In addition, EPA will post, on the WET Web site, corrections to errata in the "Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" manual (EPA 2010e.)

6. EPA is proposing to add a sentence to footnote 4 of Table II to clarify the sample holding time for the Whole Effluent Toxicity (WET) samples for the three toxicity methods (Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (5th Edition, October 2002), Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (4th Edition, October 2002), and Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms (3rd Edition, October 2002) to indicate that one sample of the minimum of three required samples may be used for the renewal of the test solutions and that the sample holding time refers to first use of each sample collected for the toxicity test. The sentence to be added is, "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."

G. Proposed Revisions to 40 CFR 136.4 and 136.5

EPA is proposing to revise §§ 136.4 and 136.5 to describe the procedures for obtaining review and EPA approval for the use of alternate test procedures (alternate methods or ATPs). The proposed changes would revise 40 CFR 136.4 to establish the procedures for obtaining approval for nationwide use of an ATP. The proposed changes would modify 40 CFR 136.5 to establish the procedures for obtaining approval for use of an ATP in a State within a particular EPA Region. It should be noted that in its ATP program, EPA considers for review only those methods for which EPA has published an ATP protocol. Presently, EPA has published protocols for chemistry, radiochemical, and culture microbiological methods. EPA does not have ATP protocols for Whole Effluent Toxicity (WET) methods or genetic methods.

In today's rule, EPA proposes to clarify that the intent of the limited use authority is to allow limited use of an alternate method for a specific application at a facility or type of discharge without requiring the same level of supporting test data that would be required for approval for nationwide use. Thus, limited use authority is not intended to be used as a means of avoiding the full examination of comparability that is required when EPA considers a method for nationwide use and decides to amend its list of approved CWA methods at 40 CFR part 136 to include alternative test procedures. In the event that EPA decides not to approve an application for approval of an alternate method for nationwide use, the Regional Alternate Test Procedures Coordinator may choose to reconsider any previous limited use approvals of the alternate method. Based on this reconsideration, the Regional Coordinator will notify the user, if the limited use approval is withdrawn.

H. Proposed Revisions to Method Modification Provisions at 40 CFR 136.6

EPA encourages regulatory authorities to allow analysts the flexibility to modify CWA methods without prior approval provided the user has documented equivalent or better performance of the method in the matrix type to which the user will apply the modified method. EPA recognizes that addressing specific matrix interferences may require modifications to approved methods that do not require the extensive review and approval process specified for an alternate test procedure at 136.4 and 136.5. Based on users' experiences with 136.6, since it was promulgated on March 12, 2007 (72 FR 11199), EPA proposes to revise this section to provide more examples of allowed and prohibited method modifications. Acceptable reasons for an analyst to modify a method include analytical practices that lower detection limits, improve precision, reduce interferences, lower laboratory costs, and promote environmental stewardship by reducing generation of laboratory wastes. Acceptable modifications may use existing or emerging analytical technologies that achieve these ends provided that they do not depart substantially from the underlying chemical principles employed in methods currently approved in 40 CFR part 136. Analysts may use the examples in this section to assess and document that their modification is acceptable and does not depart substantially from the chemical principles in the method being modified. EPA specifically invites comment on the examples of flexibility specified at 136.6 and the documentation that a method modifier must have to demonstrate the equivalency of the modified method. In particular, EPA is interested in public comment on what additional controls, if any, should be applied when changing pH, purge times, buffers, or applying the relative standard error calibration alternative.

I. Proposed New Quality Assurance and Quality Control Language at 40 CFR 136.7

EPA is proposing to specify "essential" quality control at § 136.7 for use in conducting an analysis with an approved method and when insufficient instructions are contained in an approved method. Auditors, co-regulators, laboratory personnel, and the regulated community have noted the different amounts and types of quality assurance (QA) and quality control (QC) procedures practiced by laboratories that use 40 CFR part 136 methods. Some of these methods are published by voluntary consensus standards bodies, such as the Standard Methods Committee, and ASTM International. ASTM and Standard Methods are contained in printed compendium volumes, electronic compendium volumes, or as individual online files. Each organization has its unique compendium structure. QA and QC method guidance or requirements may be listed directly in the approved consensus method, or, as is more often the case, these requirements are listed in other parts of the compendium. For example, the publisher of *Standard Methods for the Examination of Water and Wastewater* consolidates the general quality assurance and quality control requirements for all methods. Each specific Part and section can contain additional QA and QC requirements (for example, see part 2020, 3020, 6020, and 9020). ASTM specifies QA and QC requirements in the analyte method's Referenced Documents section and in the analyte method. Both organizations require the analyst to reference this additional information within the respective compendiums to achieve the QA and QC expected for valid results.

Regardless of the publisher, edition or source of an analytical method approved for CWA compliance monitoring, analysts must use suitable QA/QC procedures whether EPA or other method publishers have specified these procedures in a specific part 136 method, or referenced these procedures by other means. Consequently, EPA expects that an analyst using these consensus body methods for reporting under the CWA will also comply with the quality assurance and quality control requirements listed in the appropriate sections in the consensus body compendium. EPA's approval of use of these voluntary consensus standard body methods contemplated that any analysis using such methods would also meet the quality assurance and quality control requirements prescribed for the particular method. Thus, not following the applicable and appropriate quality assurance and quality control requirements of the respective method means that the analysis would not comply with the requirements in EPA's NPDES regulations to monitor in accordance with the procedures of 40 CFR part 136 for analysis of pollutants.

For methods that have insufficient QA/QC requirements, analysts could refer to and follow the QC published in several public sources. Examples of these sources include the instructions in an equivalent approved EPA method or standards published by the National Environmental Laboratory Accreditation Conference (cf. Chapter 5 of the compendium published in 2003.)

In addition to and regardless of the source of the laboratory's QA and QC instructions, EPA is proposing at 136.7 to specify twelve essential quality control checks that must be in the laboratory's documented quality system unless a written rationale is provided to explain why these controls are inappropriate for a specific analytical method or application. This written rationale will be included in the laboratory's Standard Operating Procedure (SOP) for each method to which specific controls do not apply (*e.g.,* internal standards, surrogate standards or tracers do not apply to analyses of inorganic parameters) as well as being included with the monitoring data produced using each method. These twelve essential quality control checks must be clearly documented in the written SOP (or method) along with a performance specification or description for each of the twelve checks.

J. Proposed Withdrawal of Appendices at 40 CFR 136

EPA is proposing to incorporate by reference all of the methods printed in 40 CFR part 136 appendices A and C, and to remove most of the information in Appendix D. EPA is proposing to remove EPA Method numbers 601 through 613, 624, 625, 1613B, 1624B and 1625B from Appendix A. All of these methods are readily accessible from a variety of sources including EPA's CWA methods Web site *http://www.epa.gov/waterscience/methods/.* Removing this appendix would decrease the resources associated with the annual publication of 40 CFR part 136 regulations. EPA would incorporate these methods by reference in Tables IC and ID at 136.3(a).

EPA is proposing to remove Appendix C—Method 200.7 Inductively Couple Plasma—Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Waste Method because this method has been superseded by Rev. 5.4 of Method 200.7, which is incorporated by reference in Table IB.

Finally, EPA is proposing to remove from Appendix D the data for all EPA methods that are no longer approved. This would result in Appendix D containing Precision and Recovery Statements only for EPA Method 279.2 for thallium and EPA Method 289.2 for zinc. EPA will correct any typographical errors in the Appendix, such as the misspelling of thallium. EPA requests comment on whether to publish and make available, at least temporarily, the current version of Appendix D online at the CWA methods Web site for historical purposes.

K. Proposed Revisions at 40 CFR 423

EPA is proposing two changes to part 423, Steam Electric Power Generating Point Source Category. First, EPA proposes to revise the definitions for *total residual chlorine* and *free available chlorine* at §§ 423.11(a) and 423.11(l), respectively. The current definitions restrict the permittee to the use of the specific amperometric titration method cited in the definitions. The revised definitions will allow the permittee flexibility to use additional approved methods. EPA proposes to revise the definitions as follows:

a. The term *total residual chlorine* (or total residual oxidants for intake water with bromides) means the value obtained using any of the "chlorine—total residual" methods in Table IB 136.3(a), or other methods approved by the permitting authority.

b. The term *free available chlorine* means the value obtained using any of the "chlorine—free available" methods in Table IB 136.3(a) where the method has the capability of measuring free available chlorine, or other methods approved by the permitting authority.

Second, EPA is proposing to move the current citations of methods from Part 423 and reference a new parameter, "chlorine-free available", in Table IB at 136.3(a). Under this parameter, EPA will list any Part 136 methods for total residual chlorine that also provide instructions for determining free chlorine. The tables at 136.3 are well known as the source of most methods that are approved for CWA programs. For this reason EPA is proposing to move the citations of specific methods from part 423 to Table IB, and as described in the following sections, also for Parts 430 and 435.

L. Proposed Revisions at 40 CFR 430

EPA is proposing several editorial changes to 40 CFR part 430, The Pulp, Paper, and Paperboard Point Source Category. Currently the complete text of EPA Methods 1650 and 1653 are published in Appendix A of part 430. EPA is proposing to cite these two methods in Table IC, at § 136.3, and to incorporate by reference the full text of these methods. EPA will list these two methods in Table IC—List of Approved Test Procedures for Non-Pesticide Organic Compounds, under adsorbable organic halides (AOX) by Method 1650 and chlorinated phenolics by Method 1653. This action would remove Appendix A at 40 CFR part 430, and organize the analytical methods for the Pulp, Paper, and Paperboard category into one part, the Part 136 CWA methods tables, of the CFR.

To help users more readily identify approved compliance monitoring methods, EPA is proposing to cite at part 430 the Part 136 methods that are approved for these pollutants: Chloroform, 2,3,7,8- tetrachlorodibenzo-p-dioxin (TCDD), and 2,3,7,8-tetrachlorodibenzo-p-furan (TCDF).

M. Proposed Revisions at 40 CFR 435

EPA is proposing several changes to Part 435, Oil and Gas Extraction Point Source Category. EPA is proposing to move, and in two cases revise, the methods from 40 CFR part 435, subpart A (Offshore Subcategory) to an EPA document ("Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013), which is included in the record for this rulemaking. This proposed approach organizes the analytical methods for the Offshore Subcategory into one document and allows for easier access to the methods for this category. The following table lists the methods EPA proposes to move from Part 435 to the cited document, EPA–821–R–09–013.

EPA METHOD NUMBERS FOR OIL AND GAS EXTRACTION POINT SOURCE CATEGORY ANALYTICAL METHODS AND PRIOR CFR REFERENCES

Analytical/test method	EPA method number	Date first promulgated	Previous CFR references
Static Sheen Test	1617	1993	Subpart A, Appendix 1.
Drilling Fluids Toxicity Test	1619	1993	Subpart A, Appendix 2.
Procedure for Mixing Base Fluids With Sediments	1646	2001	Subpart A, Appendix 3.
Protocol for the Determination of Degradation of Non Aqueous Base Fluids in a Marine Closed Bottle Biodegradation Test System: Modified ISO 11734:1995.	1647	2001	Subpart A, Appendix 4.
Determination of Crude Oil Contamination in Non-Aqueous Drilling Fluids by Gas Chromatography/Mass Spectrometry (GC/MS).	1655	2001	Subpart A, Appendix 5.
Reverse Phase Extraction (RPE) Method for Detection of Oil Contamination in Non-Aqueous Drilling Fluids (NAF).	1670	2001	Subpart A, Appendix 6.
Determination of the Amount of Non-Aqueous Drilling Fluid (NAF) Base Fluid from Drill Cuttings by a Retort Chamber (Derived from API Recommended Practice 13B–2).	1674	2001	Subpart A, Appendix 7.

EPA is also proposing to incorporate additional quality assurance procedures in the marine anaerobic biodegradation analytic method (Appendix 4 of Subpart A of Part 435) and to correct some erroneous references and omissions in the method for identification of crude oil contamination (Appendix 5 of Subpart A of Part 435). EPA is proposing to include these revisions in the EPA document (EPA–821–R–09–013).

EPA promulgated the use of the marine anaerobic biodegradation analytic method (closed bottle test, ISO 11734:1995 as clarified by Appendix 4 to Subpart A of Part 435) in 2001 because it most closely modeled the ability of a drilling fluid to biodegrade anaerobically in marine environments (January 22, 2001; 66 FR 6864). Subsequent to this promulgation, EPA incorporated additional quality assurance procedures for the marine anaerobic biodegradation analytic method in the NPDES permit for the Western Gulf of Mexico (“Final NPDES General Permit for New and Existing Sources and New Dischargers in the Offshore Subcategory of the Oil and Gas Extraction Category for the Western Portion of the Outer Continental Shelf of the Gulf of Mexico,” GMG290000, Appendix B). The additional quality assurance instructions in the GMG290000 more clearly describe the sample preparation and compliance determination steps. Specifically, these additional quality assurance procedures clarify that users must only use headspace gas to determine compliance with the Part 435 effluent guidelines.

Additionally, EPA is proposing to correct some erroneous references and omissions in the method for identification of crude oil contamination (Appendix 5 of Subpart A of Part 435). Specifically, EPA is proposing to:

a. Add a schematic flow for qualitative identification of crude oil, which was erroneously omitted in Appendix 5 to Subpart A of Part 435,

b. Correct erroneous citations in sections 9.5, 9.6, 11.3, and 11.3.1 of Appendix 5, and

c. Add a missing “<” sign for identification of crude oil contamination in the asphaltene crude discussion at Section 11.5.4.2. The asphaltene discussion now reads as follows: “Asphaltene crude oils with API gravity < 20 may not produce chromatographic peaks strong enough to show contamination at levels of the calibration. Extracted ion peaks should be easier to see than increased intensities for the C8 to C13 peaks. If a sample of asphaltene crude from the formation is available, a calibration standard shall be prepared.”

As previously noted, EPA is proposing to include these revisions to these two methods in the EPA document (EPA–821–R–09–013), which is included in the record for this rulemaking.

III. Statutory and Executive Order Reviews

A. Executive Order 12866: Regulatory Planning and Review

This rule is not a “significant regulatory action” under the terms of Executive Order (EO) 12866 (58 FR 51735, October 4, 1993) and is therefore not subject to review under the EO.

B. Paperwork Reduction Act

This action does not impose an information collection burden under the provisions of the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.* Burden is defined at 5 CFR 1320.3(b). This rule does not impose any information collection, reporting, or recordkeeping requirements. This rule merely adds new and updated versions of testing procedures, and sample preservation requirements.

C. Regulatory Flexibility Act

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

For purposes of assessing the impacts of this rule on small entities for methods under the Clean Water Act, small entity is defined as: (1) A small business that meets RFA default definitions (based on SBA size standards) found in 13 CFR 121.201; (2) a small governmental jurisdiction that is a government of a city, county, town, school district or special district with a population less than 50,000; and (3) a small organization that is any not-for-profit enterprise which is independently owned and operated and is not dominant in its field.

After considering the economic impacts of today’s proposed rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. This action approves new and updated versions of testing procedures. Generally, these changes will have a positive impact on small entities by increasing method flexibility, thereby allowing entities to reduce costs by choosing more cost-effective methods. In some cases, analytical costs may increase slightly due to the additional QC requirements included in the methods that are being approved to replace older EPA methods. However, most laboratories that analyze samples for EPA compliance monitoring have already instituted QC requirements as part of their laboratory practices.

We continue to be interested in the potential impacts of the proposed rule on small entities and welcome comments on issues related to such impacts.

D. Unfunded Mandates Reform Act

This action contains no Federal mandates under the provisions of Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), 2 U.S.C. 1531–1538 for State, local, or tribal governments, or the private sector.

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

E. Executive Order 13132: Federalism

This proposed rule does not have federalism implications. It will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132 (64 FR 43255, Aug. 10, 1999). This proposed rule merely approves new and updated versions of testing procedures. The costs to State and local governments will be minimal (in fact, governments may see a cost savings), and the rule does not preempt State law. Thus, Executive Order 13132 does not apply to this rule.

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

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, (65 FR 67249, Nov. 9, 2000). It will not have substantial direct effects on Tribal governments, on the relationship between the Federal government and Indian tribes, or on the distribution of power and responsibilities between the Federal government and Indian tribes. This rule merely approves new and updated versions of testing procedures. The costs to Tribal governments will be minimal (in fact, governments may see a cost savings), and the rule does not preempt State law. Thus, Executive Order 13175 does not apply to this rule.

In the spirit of Executive Order 13175, and consistent with EPA policy to promote communications between EPA and Indian tribes, EPA specifically solicits comment on this proposed action from tribal officials.

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

EPA interprets EO 13045 (62 FR 19885, April 23, 1997) as applying only to those regulatory actions that concern health or safety risks, such that the analysis required under section 5–501 of the EO has the potential to influence the regulation. This action is not subject to EO 13045 because it does not establish an environmental standard intended to mitigate health or safety risks. This action proposes to approve new and updated versions of testing procedures.

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

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

I. National Technology Transfer and Advancement Act of 1995

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

This proposed rulemaking involves technical standards. As described throughout this document, EPA is proposing many standards developed by the Standard Methods Committee, and ASTM International. In Sections IIB, IIC of this preamble, and the tables at § 136.3, EPA specifies these proposed methods, provides information on how to obtain copies of these methods, and describes the rationale for employing these methods. EPA welcomes comments on this aspect of the proposed rulemaking and, specifically, invites the public to identify potentially applicable voluntary consensus standards and to explain why EPA should include such standards in future revisions to Part 136.

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

Executive Order (EO) 12898 (59 FR 7629 (Feb. 16, 1994)) establishes federal executive policy on environmental justice. Its main provision directs federal agencies, to the greatest extent practicable and permitted by law, to make environmental justice part of their mission by identifying and addressing, as appropriate, disproportionately high and adverse human health or environmental effects of their programs, policies, and activities on minority populations and low-income populations in the United States.

This proposed rule provides additional compliance methods for use by any facility or laboratory with no disproportionate impact on minority or low-income populations because it merely proposes to approve new and updated versions of testing procedures to measure pollutants in water.

IV. References

EPA 2006, "Assessment of the Effects of Holding Time on Enterococci Concentrations in Fresh and Marine Recreational Waters and *Escherichia coli* Concentrations in Fresh Recreational Waters" (EPA–821–R–06–019, December 2006)

EPA 2010a, "Method 1668A Interlaboratory Validation Study Report" (EPA–820–R–10–004, March 2010)

EPA 2010b, "Addendum to the Method 1668A Interlaboratory Validation Study Report" (EPA–820–R–10–003, March 2010)

EPA 2010c, "Peer Review of the Method 1668A Interlaboratory Validation Study" (EPA 820–R–10–007, April 2010)

EPA 2010d, "Development of Pooled Method Detection Limits (MDLs) and Minimum Levels of Quantitation (MLs) for EPA Method 1668C (May 2010)

EPA 2010e, Errata for "Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (4th edition, October 2002) manual.

OSS 2009, ASTM D7575 "Inter-Laboratory Study to Establish Precision Statements for ASTM WK23240—Standard Test Method for Solvent-Free Membrane Recoverable Oil and Grease by Infrared Determination"
Passaic River 2010 "Summary of Passaic River Split Sample Results", EPA, April 2010
Test America 1 "Acrolein Acrylonitrile Stability Study"
Test America 2 "Acrolein Acrylonitrile Control Charts"

List of Subjects

40 CFR Part 136

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

40 CFR Part 260

Environmental protection, Administrative practice and procedure, Confidential business information, Hazardous waste, Incorporation by reference, Reporting and recordkeeping requirements.

40 CFR Part 423

Environmental protection, Steam Electric Power Generating Point Source Category, Incorporation by reference, Reporting and recordkeeping requirements, Water pollution control.

40 CFR Part 430

Environmental protection, Pulp, Paper, and Paperboard Point Source Category, Incorporation by reference, Reporting and recordkeeping requirements, Water pollution control.

40 CFR Part 435

Environmental protection, Oil and Gas Extraction Point Source Category, Incorporation by reference, Reporting and recordkeeping requirements, Water pollution control.

Dated: August 6, 2010.

Lisa P. Jackson,

Administrator.

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

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

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

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

2. Section 136.1 is amended by revising paragraph (a) to read as follows:

§ 136.1 Applicability.

(a) The procedures prescribed herein shall, except as noted in §§ 136.4, 136.5, and 136.6, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(1) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of title 40; and

(2) Reports required to be submitted by dischargers under the NPDES established by parts 124 and 125 of this chapter; and

(3) Certifications issued by States pursuant to section 401 of the CWA, as amended.

* * * * *

3. Section 136.3 is amended:

a. By revising paragraph (a) introductory text;

b. In paragraph (a), revise Table IA, IB, IC, ID, IG, and IH;

c. By revising paragraphs (b)(1), (b)(54), (b)(55), (b)(56), (b)(59), (b)(60), (b)(61), (b)(70), and adding paragraph (b)(73);

d. By revising paragraph (e) introductory text;

e. In Table II to paragraph (e), by revising entries "Table IA—Bacterial Tests", "Table IA—Aquatic Toxicity Tests", "Table IH—Bacterial Tests", and "Table IH—Protozoan Tests, and footnote 6".

These 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, and IH. In the event of a conflict between the reporting requirements of 40 CFR parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of 40 CFR Parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG, and IH. The incorporation by reference of these documents, as specified in paragraph (b) of this section, was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies of the documents may be obtained from the sources listed in paragraph (b) of this section. Documents may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue, NW., Room 3334, Washington, DC (Telephone: 202–566–2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.* These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the **Federal Register**. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, IE, IF, IG, and IH 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. Under certain circumstances paragraph (c) of this section, § 136.5 or 40 CFR 401.13, other additional or alternate test procedures may be used.

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

Parameter and units	Method[1]	EPA	Standard methods	AOAC, ASTM, USGS	Other
Bacteria:					
1. Coliform (fecal), number per 100 mL or number per gram dry weight.	Most Probable Number (MPN), 5 tube, 3 dilution, or	p. 132[3] .... 1680[11, 13] 1681[11, 18]	9221 C E–2006.		

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

Parameter and units	Method[1]	EPA	Standard methods	AOAC, ASTM, USGS	Other
	Membrane filter (MF)[2], single step.	p. 124[3]	9222 D–1997	B–0050–85.[4]	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132[3]	9221 C E–2006.		
	MF[2], single step	p. 124[3]	9222 D–1997.		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B–2006.		
	MF[2], single step or two step.	p. 108[3]	9222 B–1997	B–0025–85.[4]	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B–2006.		
	MF[2] with enrichment	p. 111[3]	9222 (B+B.5c) – 1997.		
5. E. coli, number per 100 mL.[19]	MPN[6,8,14] multiple tube/multiple well.		9223 B–2004[12]	991.15[10]	Colilert®[12,16] Colilert-18®.[12,15,16]
	MF[2,5,6,7,8] single step.	1603[20]			mColiBlue-24®.[17]
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution,	p. 139[3]	9230 B–2007.		
	MF[2], or	p. 136[3]	9230 C–2007	B–0055–85.[4]	
	Plate count	p. 143.[3]			
7. Enterococci, number per 100. mL.[19]	MPN[6 8], multiple tube/multiple well.			D6503–99[9]	Enterolert®.[12 22]
	MF[2 5 6 7 8] single step	1600.[23]			
8. Salmonella, number per gram dry. weight.[11]	MPN multiple tube	1682.[21]			
Aquatic Toxicity:					
9. Toxicity, acute, fresh water organisms, LC_{50}, percent effluent.	*Ceriodaphnia dubia* acute.	2002.0.[24]			
	Daphnia puplex and *Daphnia magna* acute.	2021.0.[24]			
	Fathead minnow, *Pimephales promelas,* and Bannerfin shiner, *Cyprinella leedsi,* acute.	2000.0.[24]			
	Rainbow Trout, *Oncorhynchus mykiss,* and brook trout, *Salvelinus fontinalis,* acute.	2019.0.[24]			
10. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC_{50}, percent effluent.	Mysid, *Mysidopsis bahia,* acute.	2007.0.[24]			
	Sheepshead Minnow, *Cyprinodon variegatus,* acute.	2004.0.[24]			
	Silverside, *Menidia beryllina, Menidia menidia,* and *Menidia peninsulae,* acute.	2006.0.[24]			

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

Parameter and units	Method [1]	EPA	Standard methods	AOAC, ASTM, USGS	Other
11. Toxicity, chronic, fresh water organisms, NOEC or IC_{25}, percent effluent.	Fathead minnow, *Pimephales promelas,* larval survival and growth.	1000.0.[25]			
	Fathead minnow, *Pimephales promelas,* embryo-larval survival and teratogenicity.	1001.0.[25]			
	Daphnia, *Ceriodaphnia dubia,* survival and reproduction.	1002.0.[25]			
	Green alga, *Selenastrum capricornutum,* growth.	1003.0.[25]			
12. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC_{25}, percent effluent.	Sheepshead minnow, *Cyprinodon variegatus,* larval survival and growth.	1004.0.[26]			
	Sheepshead minnow, *Cyprinodon variegatus,* embryo-larval survival and teratogenicity.	1005.0.[26]			
	Inland silverside, *Menidia beryllina,* larval survival and growth.	1006.0.[26]			
	Mysid, *Mysidopsis bahia,* survival, growth, and fecundity.	1007.0.[26]			
	Sea urchin, *Arbacia punctulata,* fertilization.	1008.0.[26]			

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

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

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

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

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

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

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

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

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

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

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

[12] These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by *E. coli.*

[13] USEPA. April 2010. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–10–003.

[14] Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray®, Quanti-Tray®/2000, and the MPN calculated from the table provided by the manufacturer.

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

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

[17] A description of the mColiBlue24® test, is available from Hach Company. 100 Dayton Ave., Ames, IA 50010.

[18] USEPA. July 2006. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A–1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–06–013.

[19] Recommended for enumeration of target organism in wastewater effluent.

[20] USEPA. December 2009. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–09–007.

[21] USEPA. July 2006. Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–06–014.

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

[23] USEPA. December 2009. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-*Enterococcus* Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC, EPA–821–R–09–016.

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

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

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

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
1. Acidity, as CaCO$_3$, mg/L.	Electrometric endpoint or phenolphthalein endpoint.		2310 B–1997	D1067–06	I–1020–85.[2]
2. Alkalinity, as CaCO$_3$, mg/L.	Electrometric or Colorimetric titration to pH 4.5, Manual.		2320 B–1997	D1067–06	973.43,[3] I–1030–85.[2]
	Automatic	310.2 (Rev. 1974)[1]			I–2030–85.[2]
3. Aluminum—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 D–1999 or E–1999.		I–3051–85.[2]
	AA furnace		3113–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	Direct Current Plasma (DCP)[36].			D4190–08	See footnote.[34]
	Colorimetric (Eriochrome cyanine R).		3500–Al B–2001.		
4. Ammonia (as N), mg/L.	Manual distillation[6] or gas diffusion (pH > 11) followed by any of the following:	350.1, Rev. 2.0 (1993).	4500–NH$_3$ B–1997		973.49.[3]
	Nesslerization			D1426–08 (A)	973.49,[3] I–3520–85.[2]
	Titration		4500–NH$_3$ C–1997.		
	Electrode		4500–NH$_3$ D–1997 or E–1997.	D1426–08 (B).	
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.		4500–NH$_3$ F–1997		See Footnote.[60]
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.	350.1,[30] Rev. 2.0 (1993).	4500–NH$_3$ G–1997 4500–NH$_3$ H–1997.		I–4523–85.[2]
	Automated electrode				See footnote 7.
	Ion Chromatography			D6919–09.	
5. Antimony—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 B–1999.		
	AA furnace		3113–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
6. Arsenic—Total,[4] mg/L.	Digestion[4] followed by any of the following:	206.5 (Issued 1978).[1]			
	AA gaseous hydride		3114 B–2009 or C–2009.	D2972–08 (B)	I–3062–85.[2]
	AA furnace		3113–2004	D2972–08 (C)	I–4063–98.[49]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05.
	Colorimetric (SDDC)		3500–As B–1997	D2972–08 (A)	I–3060–85.[2]
7. Barium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 D–1999		I–3084–85.[2]
	AA furnace		3113–2004	D4382–02(07).	
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP[36]				See footnote.[34]
8. Beryllium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 D–1999 or E–1999.	D3645–08 (A)	I–3095–85.[2]
	AA furnace		3113–2004	D3645–08 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP			D4190–08	See footnote.[34]
	Colorimetric (aluminon).				See footnote.[61]
9. Biochemical oxygen demand (BOD5), mg/L.	Dissolved Oxygen Depletion.		5210 B–2001	D888–09	973.44,[3] p. 17,[9] I–1578–78,[8] See footnote.[10] [63]
10. Boron—Total,[37] mg/L.	Colorimetric (curcumin)		4500–B B–2000		I–3112–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP			D4190–08	See footnote.[34]
11. Bromide, mg/L	Electrode			D1246–05	I–1125–85.[2]
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000, C–2000, D–2000.	D4327–03	993.30.[3]
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
12. Cadmium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 B–1999 or C–1999.	D3557–02(07) (A or B).	974.27,[3] p. 37.[9], I–3135–85[2] or I–3136–85.[2]
	AA furnace		3113–1999	D3557–02(07) (D)	I–4138–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–1472–85[2] or I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP[36]			D4190–08	See footnote.[34]
	Voltametry[11]			D3557–02(07)(C).	

Table IB—List of Approved Inorganic Test Procedures—Continued

Parameter	Methodology [58]	EPA [52]	Standard methods	ASTM	USGS/AOAC/other
	Colorimetric (Dithizone)		3500 Cd–D 1990.		
13. Calcium—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration		3111 B–1999	D511–08(B)	I–3152–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
	DCP				See footnote.[34]
	Titrimetric (EDTA)		3500–Ca–1997	D511–08(A).	
	Ion Chromatography			D6919–09.	
14. Carbonaceous biochemical oxygen demand (CBOD5), mg/L.[12]	Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B–2001	D888–09	See footnote.[35] [63]
15. Chemical oxygen demand (COD), mg/L.	Titrimetric	410.3 (Rev. 1978) [1]	5220 B–1997 or C–1997.	D1252–06 (A)	973.46,[3] p. 17 [9] I–3560–85.[2]
	Spectrophotometric, manual or automatic.	410.4, Rev. 2.0 (1993).	5220 D–1997	D1252–06 (B)	See footnotes.[13] [14] I–3561–85.[2]
16. Chloride, mg/L	Titrimetric: (silver nitrate)		4500–Cl^- B–1997	D512–04 (B)	I–1183–85.[2]
	(Mercuric nitrate)		4500–Cl^- C–1997	D512–04 (A)	973.51,[3] I–1184–85.[2]
	Colorimetric: manual				I–1187–85.[2]
	Automated (Ferricyanide)		4500–Cl^- E–1997		I–2187–85.[2]
	Potentiometric Titration		4500–Cl^- D–1997.		
	Ion Selective Electrode			D512–04 (C).	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000 or C–2000.	D4327–03	993.30,[3] I–2057–90.[51]
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
17. Chlorine—Total residual, mg/L.	Amperometric direct		4500–Cl D–2000	D1253–08.	
	Amperometric direct (low level).		4500–Cl E–2000.		
	Iodometric direct		4500–Cl B–2000.		
	Back titration ether end–point [15].		4500–Cl C–2000.		
	DPD–FAS		4500–Cl F–2000.		
	Spectrophotometric, DPD		4500–Cl G–2000.		
	Electrode				See footnote.[16]
17A. Chlorine—Free Available, mg/L.	Amperometric direct		4500–Cl D–2000	D1253–08.	
	Amperometric direct (low level).		4500–Cl E–2000.		
	DPD–FAS		4500–Cl F–2000.		
	Spectrophotometric, DPD		4500–Cl G–2000.		
18. Chromium VI dissolved, mg/L.	0.45-micron Filtration followed by any of the following:				
	AA chelation-extraction.		3111 C–1999		I–1232–85.[2]
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500–Cr C–2009	D5257–03	993.23.
	Colorimetric (Diphenyl-carbazide).		3500–Cr B–2009	D1687–02(07)(A)	I–1230–85.[2]
19. Chromium—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration [36].		3111 B–1999	D1687–02(07) (B)	974.27,[3] I–3236–85.[2]
	AA chelation-extraction.		3111 C–1999.		
	AA furnace		3113–1999	D1687–02(07)(C)	I–3233–93.[46]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES [36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05.
	DCP [36]			D4190–08	See footnote.[34]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	EPA [52]	Standard methods	ASTM	USGS/AOAC/other
	Colorimetric (Diphenyl-carbazide).		3500–Cr B–2009.		
20. Cobalt—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration		3111 B–1999 or C–1999.	D3558–08 (A or B)	p. 37,[9] I–3239–85.[2]
	AA furnace		3113–2004	D3558–08 (C)	I–4243–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05.
	DCP			D4190–08	See footnote.[34]
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity.	Colorimetric (ADMI)				See footnote.[18]
	(Platinum cobalt) Spectrophotometric.		2120 B–2001		I–1250–85.[2]
22. Copper—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration.[36]		3111 B–1999 or C–1999.	D1688–07 (A or B)	974.27 [3] p. 37 [9] I–3270–85 [2] or I–3271–85.[2]
	AA furnace		3113–2004	D1688–07 (C)	I–4274–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES [36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05
	DCP [36]			D4190–08	See footnote.[34]
	Colorimetric (Neocuproine).		3500–Cu B–1999.		
	(Bathocuproine)		3500–Cu C–1999		See footnote.[19]
23. Cyanide—Total, mg/L.	Automated UV digestion/distillation and Colorimetry.				Kelada–01.[55]
	Segmented Flow Injection, In-Line Ultraviolet Digestion followed by gas diffusion amperometry.			D7511–09e2.	
	Manual distillation with $MgCl_2$ followed by any of the following:	335.4, Rev. 1.0 (1993) [57].	4500–CN^- B–1999 or C–1999.	D2036–09(A), D7284–08.	10–204–00–1–X.[56]
	Flow Injection, gas diffusion amperometry.			D2036–09(A) D7284–08.	
	Titrimetric		4500–CN^- D–1999	D2036–09(A)	p. 22.[9]
	Spectrophotometric, manual.		4500–CN^- E–1999	D2036–09(A)	I–3300–85.[2]
	Semi-Automated [20]	335.4, Rev. 1.0 (1993) [57].			10–204–00–1–X,[56] I–4302–85.[2]
	Ion Chromatography			D2036–09(A).	
	Ion Selective Electrode.		4500–CN^- F–1999	D2036–09(A).	
24. Cyanide-Available, mg/L.	Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$ followed by Titrimetric or Spectrophotometric.		4500–CN^- G–1999	D2036–09(B).	
	Flow injection and ligand exchange, followed by gas diffusion amperometry [59].			D6888–09	OIA–1677–09.[44]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	Automated Distillation and Colorimetry (no UV digestion).				Kelada–01.[55]
24.A Cyanide-Free, mg/L.	Flow Injection, followed by gas diffusion amperometry.			D7237–10	OIA–1677–09.[44]
	Manual micro-diffusion and colorimetry.			D4282–02.	
25. Fluoride—Total, mg/L.	Manual distillation[6] followed by any of the following:		4500–F$^-$ B–1997.		
	Electrode, manual		4500–F$^-$ C–1997	D1179–04(B).	
	Electrode, automated.				I–4327–85.[2]
	Colorimetric, (SPADNS).		4500–F$^-$ D–1997	D1179–04(A).	
	Automated complexone.		4500–F$^-$ E–1997.		
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000 or C–2000.	D4327–03	993.30.[3]
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
26. Gold—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration,		3111 B–1999.		
	AA furnace,	231.2 (Rev. 1978)[1]	3113–2004.		
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
	DCP				See footnote.[34]
27. Hardness—Total, as CaCO3, mg/L.	Automated colorimetric,	130.1 (Issued 1971)[1].			
	Titrimetric (EDTA)		2340 C–1997	D1126–02(07)	973.5 2B,[3] I–1338–85.[2]
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).		2340 B–1997.		
28. Hydrogen ion (pH), pH units.	Electrometric measurement.		4500–H$^+$–2000	D1293–99 (A or B)	973.41,[3] I–1586–85.[2]
	Automated electrode	150.2 (Dec. 1982)[1]			See footnote,[21] I–2587–85.[2]
29. Iridium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 B–1999.		
	AA furnace	235.2 (Issued 1978).[1]			
	ICP/MS		3125–2009.		
30. Iron—Total,[4] mg/L	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 B–1999 or C–1999.	D1068–05 (A or B)	974.27,[3] I–3381–85.[2]
	AA furnace		3113–1999	D1068–05(C).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
	DCP[36]			D4190–08	See footnote.[34]
	Colorimetric (Phenanthroline).		3500–Fe-1997	D1068–05 (D)	See footnote.[22]
31. Kjeldahl Nitrogen[5]—Total, (as N), mg/L.	Manual digestion[20] and distillation or gas diffusion followed by any of the following:		4500–Norg B–1997 or C–1997 and 4500–NH$_3$ B–1997.	D3590–02(06)(A)	I–4515–91.[45]
	Titration		4500–NH$_3$ C–1997		973.48.[3]
	Nesslerization			D1426–08(A).	
	Electrode		4500–NH$_3$ D–1997 or E–1997.	D1426–08(B).	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	Semi-automated phenate.	350.1 Rev. 2.0 1993	4500–NH_3 G–1997 4500–NH_3 H–1997.		
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.		4500–NH_3 F–1997		See Footnote.[60]
	Automated Methods for TKN that do not require manual distillation				
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation).	351.1, (Rev. 1978)[1]			I–4551–78.[8]
	Semi-automated block digestor colorimetric (distillation not required).	351.2, Rev. 2.0 (1993).	4500–Norg D–1997	D3590–02(06) (B)	I–4515–91.[45]
	Block digester, followed by Auto distillation and Titration.				See footnote.[39]
	Block digester, followed by Auto distillation and Nesslerization.				See footnote.[40]
	Block Digester, followed by Flow injection gas diffusion (distillation not required).				See footnote.[41]
32. Lead—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration.[36]		3111 B–1999 or C–1999.	D3559–08(A or B)	974.27,[3] I–3399–85.[2]
	AA furnace		3113–1999	D3559–08(D)	I–4403–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP[36]			D4190–08	See footnote.[34]
	Voltametry[11]			D3559–08(C).	
	Colorimetric (Dithizone).		3500–Pb B–1997.		
33. Magnesium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 B–1999	D511–08(B)	974.27,[3] I–3447–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
	DCP				See footnote.[34]
	Gravimetric.				
	Ion Chromatography			D6919–09.	
34. Manganese—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 B–1999	D858–07(A or B)	974.27,[3] I–3454–85.[2]
	AA furnace		3113–2004	D858–07(C).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP[36]			D4190–08	See footnote.[34]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	Colorimetric (Persulfate).		3500–Mn B–1999		920.203.[3]
	(Periodate)				See footnote.[23]
35. Mercury—Total,[4] mg/L.	Cold vapor, Manual	245.1, Rev. 3.0 (1994).	3112–2009	D3223–07	977.22,[3] I–3462–85.[2]
	Cold vapor, Automated	245.2 (Issued 1974).			
	Cold vapor atomic fluorescence spectrometry (CVAFS).	245.7, Rev. 2.0 (2005)[17].			I–4464–01.
	Purge and Trap CVAFS	1631E.[43]			
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120–1999		I–4471–97.[50]
	ICP/MS		3125–2009.		
36. Molybdenum—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 D–1999		I–3490–85.[2]
	AA furnace		3113–2004		I–3492–96.[47]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP				See footnote.[34]
37. Nickel—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].		3111 B–1999 or C–1999.	D1886–08(A or B)	I–3499–85.[2]
	AA furnace		3113–2004	D1886–08(C)	I–4503–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05.
	DCP[36]			D4190–08	See footnote.[34]
38. Nitrate (as N), mg/L.	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000 or C–2000.	D4327–03	993.30.[3]
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
	Ion Selective Electrode		4500–NO_3^- D–2000.		
	Colorimetric (Brucine sulfate).	352.1[1]			973.50,[3] 419D,[1 7] p. 28.[9]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).				See footnote.[62]
39. Nitrate-nitrite (as N), mg/L.	Cadmium reduction, Manual.		4500–NO_3^- E–2000	D3867–04(B).	
	Cadmium reduction, Automated.	353.2, Rev. 2.0 (1993).	4500–NO_3^- F–2000	D3867–04(A)	I–2545–90.[2]
	Automated hydrazine		4500–NO_3^- H–2000.		
	Reduction/Colorimetric				See footnote.[62]
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000 or C–2000.	D4327–03	993.30.[3]
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
40. Nitrite (as N), mg/L	Spectrophotometric: Manual.		4500–NO_2^- B–2000		See footnote.[25]
	Automated (Diazotization).				I–4540–85,[2] See footnote.[62]
	Automated (*bypass cadmium reduction).	353.2, Rev. 2.0 (1993).	4500–NO_3^- F–2000	D3867–04 (A)	I–4545–85.[2]
	Manual (*bypass cadmium reduction).		4500–NO_3^- E–2000	D3867–04 (B).	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000 or C–2000.	D4327–03	993.30.[3]
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev.2.[54]
41. Oil and grease—Total recoverable, mg/L.	Hexane extractable material (HEM): n-Hexane extraction and gravimetry.	1664B[42]	5520 B–2001.[38]		

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	EPA [52]	Standard methods	ASTM	USGS/AOAC/other
	Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry.	1664B [42]	5520 B–2001 [38] and 5520 F–2001.[38]		
42. Organic carbon—Total (TOC), mg/L.	Combustion	...	5310 B–2000	D7573–09	973.47,[3] p. 14.[24]
	Heated persulfate or UV persulfate oxidation.	...	5310 C 2000 5310 D 2000.	D4839–03	973.47,[3] p. 14.[24]
43. Organic nitrogen (as N), mg/L.	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4).				
44. Orthophosphate (as P), mg/L.	Ascorbic acid method:				
	Automated	365.1, Rev. 2.0 (1993).	4500–P F–1999 or G–1999.	...	973.56,[3] I–4601–85.[2]
	Manual single reagent.	...	4500–P E–1999	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–2000 or C–2000.	D4327–03	993.30.[3]
	CIE/UV	...	4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
45. Osmium—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration	...	3111 D–1999.		
	AA furnace	252.2 (Issued 1978) [1]			
46. Oxygen, dissolved, mg/L.	Winkler (Azide modification).	...	4500–O B–2001, C–2001, D–2001, E–2001, F–2001.	D888–09(A)	973.45B,[3] I–1575–78.[8]
	Electrode	...	4500–O G–2001	D888–09(B)	I–1576–78.[8]
	Luminescence Based Sensor.	...	...	D888–09 [68] (C)	See footnote [63] [68] See footnote.[64]
47. Palladium—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration	...	3111 B–1999.		
	AA furnace	253.2 [1] (Issued 1978).			
	ICP/MS	...	3125–2009.		
	DCP	...	...	...	See footnote.[34]
48. Phenols, mg/L	Manual distillation [26] followed by any of the following:	420.1 [1] (Rev. 1978)	5530B–2005	D1783–01.	
	Colorimetric (4AAP) manual.	420.1 [1] (Rev. 1978)	5530D–2005 [27]	D1783–01(A or B).	
	Automated colorimetric (4AAP).	420.4, Rev. 1.0 (1993).			
49. Phosphorus (elemental), mg/L.	Gas–liquid chromatography.	...	...	...	See footnote.[28]
50. Phosphorus—Total, mg/L.	Persulfate digestion [20] followed by any of the following:	...	4500–P B(5)–1999	...	973.55.[3]
	Manual	365.3 [1] (Issued 1978).	4500–P E–1999	D515–88(A).	
	Automated ascorbic acid reduction.	365.1, Rev. 2.0 (1993).	4500–P F–1999, G–1999, H–1999.	...	973.56,[3] I–4600–85.[2]
	ICP/AES [4] [36]	200.7, Rev. 4.4 (1994).	3120–1999	...	I–4471–97.[50]
	Semi–automated block digestor (TKP digestion).	365.4 [1] (Issued 1974).	...	D515–88(B)	I–4610–91.[48]
51. Platinum—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration	...	3111 B–1999.		
	AA furnace	255.2.[1]			
	ICP/MS	...	3125–2009.		
	DCP	...	...	...	See footnote.[34]
52. Potassium—Total,[4] mg/L.	Digestion [4] followed by any of the following:				
	AA direct aspiration	...	3111 B–1999	...	973.53,[3] I–3630–85.[2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	ICP/AES	200.7, Rev. 4.4 (1994).	3120–1999.		
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
	Flame photometric		3500–K B–1997.		
	Electrode		3500–K C–1997.		
	Ion Chromatography			D6919–09.	
53. Residue—Total, mg/L.	Gravimetric, 103–105°		2540 B–1997		I–3750–85.[2]
54. Residue—filterable, mg/L.	Gravimetric, 180°		2540 C–1997	D5907–03	I–1750–85.[2]
55. Residue—non–filterable (TSS), mg/L.	Gravimetric, 103–105° post washing of residue.		2540 D–1997	D5907–03	I–3765–85.[2]
56. Residue—settleable, mg/L.	Volumetric, (Imhoff cone), or gravimetric.		2540 F–1997.		
57. Residue—Volatile, mg/L.	Gravimetric, 550°	160.4[1]	2540–E–1997		I–3753–85.[2]
58. Rhodium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration, or.		3111 B–1999.		
	AA furnace	265.2.[1]			
	ICP/MS		3125–2009.		
59. Ruthenium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration, or.		3111 B–1999.		
	AA furnace	267.2.[1]			
	ICP/MS		3125–2009.		
60. Selenium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA furnace		3113–2004	D3859–08 (B)	I–4668–98.[49]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05
	AA gaseous hydride		3114 B–2009, or C–2009.	D3859–08 (A)	I–3667–85.[2]
61. Silica—Dissolved,[37] mg/L.	0.45 micron filtration followed by any of the following:				
	Colorimetric, Manual		4500–SiO_2 C–1997	D859–05	I–1700–85.[2]
	Automated (Molybdosilicate).		4500–SiO_2 E–1997 or F–1997.		I–2700–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999		I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
62. Silver—Total,[4][31] mg/L.	Digestion[4][29] followed by any of the following:				
	AA direct aspiration		3111 B–1999 or C–1999.		974.27,[3] p. 37,[9] I–3720–85.[2]
	AA furnace		3113 –1999		I–4724–89.[51]
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
	DCP				See footnote.[34]
63. Sodium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 B–1999		973.54,[3] I–3735–85.[2]
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999		I–4471–97.[50]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
	DCP				See footnote.[34]
	Flame photometric		3500–Na B–1997.		
	Ion Chromatography			D6919–09.	
64. Specific conductance, micromhos/cm at 25 °C.	Wheatstone bridge	120.1[1] (Rev. 1982)	2510 –1997	D1125–99 (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–1997 or G–1997.		
	Gravimetric		4500–SO_4^{2-} C–1997 or D–1997.		925.54.[3]
	Turbidimetric		4500–SO_4^{2-} E–1997.	D516–07.	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2000 or C–2000.	D4327–03	993.30,[3] I–4020–05.
	CIE/UV		4140–1997	D6508–00(05)	D6508, Rev. 2.[54]
66. Sulfide (as S), mg/L.	Sample Pretreatment		4500–S^{2-} B, C–2000.		
	Titrimetric (iodine)		4500–S^{2-} F–2000		I–3840–85.[2]
	Colorimetric (methylene blue).		4500–S^{2-} D–2000.		
	Ion Selective Electrode		4500–S^{2-} G–2000	D4658–08.	
67. Sulfite (as SO_3), mg/L.	Titrimetric (iodine-iodate)		4500–SO_3^{2-} B–2000.		
68. Surfactants, mg/L	Colorimetric (methylene blue).		5540 C–2000	D2330–02.	
69. Temperature, °C	Thermometric		2550 B–2000		See footnote.[32]
70. Thallium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 B–1999.		
	AA furnace	279.2[1] (Issued 1978).	3113–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.7, Rev. 4.4 (1994).	3120–1999	D1976–07.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4471–97.[50]
71. Tin—Total,[4] mg/L	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 B–1999		I–3850–78.[8]
	AA furnace		3113–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).			
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
72. Titanium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 D–1999.		
	AA furnace	283.2[1](Issued 1978).			
	DCP				See footnote.[34]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14.[3]
73. Turbidity, NTU[53]	Nephelometric	180.1, Rev. 2.0 (1993).	2130–2001	D1889–00	I–3860–85.[2] See footnote.[65] See footnote.[66] See footnote.[67]
74. Vanadium—Total,[4] mg/L.	Digestion[4] followed by any of the following:				
	AA direct aspiration		3111 D–1999.		
	AA furnace		3113–2004	D3373–03(07).	
	ICP/AES	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05.
	DCP			D4190–08	See footnote.[34]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[58]	EPA[52]	Standard methods	ASTM	USGS/AOAC/other
	Colorimetric (Gallic Acid).	....	3500–V B–1997.		
75. Zinc—Total,[4] mg/L	Digestion[4] followed by any of the following:				
	AA direct aspiration[36].	....	3111 B–1999 or C–1999.	D1691–02(07) (A or B).	974.27,[3] p. 37,[9] I–3900–85.[2]
	AA furnace	289.2[1] (Issued 1978).			
	ICP/AES[36]	200.5, Rev. 4.2 (2003); 200.7, Rev. 4.4 (1994).	3120–1999	D1976–07	I–4471–97.[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	3125–2009	D5673–05	993.14,[3] I–4020–05.
	DCP[36]	....	....	D4190–08	See footnote.[34]
	Colorimetric (Dithizone).				
	(Zincon)	....	3500–Zn–1997	....	See footnote.[33]
76. Acid Mine Drainage.	....	1627.			

Table 1B Notes:

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

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

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

[4] For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of “Methods for the Determination of Metals in Environmental Samples” EPA/600R–94/111, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP–AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP–AES, and ICP–MS) use EPA Method 200.2 or an approved alternate procedure (*e.g.,* CEM microwave digestion, which may be used with certain analytes as indicated in Table IB); the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as “total” metals.

[5] Copper sulfate or other catalysts that have been found suitable may be used in place of mercuric sulfate.

[6] Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary: however, manual distillation will be required to resolve any controversies. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step. (A total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as <20% RPD for all tested matrices). Alternatively the two populations of spike recovery percentages may be compared using a recognized statistical test.

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

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

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

[10] In-Situ Method 1003–8–2009, “Biochemical Oxygen Demand (BOD) Measurement by Optical Probe”. Available from In-Situ, Incorporated, 221 E. Lincoln Avenue, Ft. Collins, CO 80524, Telephone: 970–498–1500.

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

[12] Carbonaceous biochemical oxygen demand (CBOD5) must not be confused with the traditional BOD5 test method which measures “total BOD.” The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD5 parameter. A discharger whose permit requires reporting the traditional BOD5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger’s permit specifically states CBOD5 is required can the permittee report data using a nitrification inhibitor.

[13] OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 151 Graham Road, P.O. Box 9010, College Station, TX 77842.

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

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

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

[17] Method 245.7, Rev. 2.0, “Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry,” February 2005, EPA–821–R–05–001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304, *Telephone:* 703–461–2100, Fax: 703–461–8056.

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

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

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

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

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

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

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

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

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

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

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

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

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

[31] For samples known or suspected to contain high levels of silver (*e.g.,* in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH4OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I2 to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH >7 with NH4OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.

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

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

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

[35] In-Situ Method 1004–8–2009, "Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe". Available from In-Situ, Incorporated, 221 E. Lincoln Avenue, Ft. Collins, CO 80524, Telephone: 970–498–1500.

[36] Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, NC 28106–0200, April 16, 1992. Available from the CEM Corporation.

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

[38] Only use n-hexane (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 Method 1664B). Use of other extraction solvents is prohibited.

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

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

[41] Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/96, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[42] Method 1664, Revision B is the revised version of EPA Method 1664A.

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

[44] Available Cyanide, Method OIA–1677–09, "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842–9010.

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

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

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

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

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

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

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

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

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

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

[55] Kelada-01, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821–B–01–009, Revision 1.2, August 2001, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. **Note:** A 450–W UV lamp may be used in this method instead of the 550–W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.

[56] QuikChem Method 10–204–00–1–X, "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis" is available from Lachat Instruments 6645 W. Mill Road, Milwaukee, WI 53218, *Telephone:* 414–358–4200.

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

[58] Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation "followed by" analysis with a method, approved digestion and/or distillation are required prior to analysis.

[59] Samples analyzed for available cyanide using Methods OIA–1677–09 or D6888–09 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample filtration to no more than 30 minutes to preclude settling of materials in samples.

[60] Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction ("phenol-hypochlorite reaction") colorimetric ammonium determination methods. For example when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, C.J. Patton and S.R. Crouch, Anal. Chem. (1977) 49, 464–469. These reaction parameters increase to pH >12.6 and 665 nm when salicylate is used as the color reagent—see, M.D. Krom, Analyst (1980) 105, 305–316.

[61] If atomic absorption or ICP instrumentation is not available, the aluminon colorimetric method detailed in the 19th Edition of *Standard Methods* may be used. This method has poorer precision and bias than the methods of choice.

[62] Systea Easy (1-Reagent) Nitrate Method, February 4, 2009. Available at *http://www.nemi.gov* or from Systea Scientific, LLC., 900 Jorie Blvd., Suite 35, Oak Brook, IL 60523.

[63] Hach Method 10360, "Luminescence Measurement of Dissolved Oxygen (LDO®) in Water and Wastewater, Revision 1.1 dated January 4, 2006". Available from Hach Company, 5600 Lindbergh Drive, Loveland, CO 80539, *Telephone:* 970–669–3050.

[64] In-Situ Method 1002–8–2009, "Dissolved Oxygen (DO) Measurement by Optical Probe", 1003–8–2009. Available from In-Situ, Incorporated, 221 E. Lincoln Avenue, Ft. Collins, CO 80524, *Telephone:* 970–498–1500.

[65] Mitchell Method M5331, "Determination of Turbidity by Nephlometry", Revision 1.0, July 31, 2008. Available from Leck Mitchell, Ph.D., P.E., 656 Independence Valley Drive, Grand Junction Colorado 81507, *Phone:* 630–645–0600.

[66] Mitchell Method M5271, "Determination of Turbidity by Nephlometry", Revision 1.0, July 31, 2008. Available from Leck Mitchell, Ph.D., P.E., 656 Independence Valley Drive, Grand Junction Colorado 81507, *Phone:* 630–645–0600.

[67] Thermo Scientific's Orion Method AQ4500, Revision 5, March 12, 2009, "Determination of Turbidity by Nephlometry". Available from Thermo Scientific, 166 Cummings Center, Beverly, MA 01915, *Phone:* 1–800–225–1480, *http://www.thermo.com.*

[68] This method may be used to measure dissolved oxygen when performing methods approved in Table 1B for measurement of biochemical oxygen demand for compliance monitoring under the Clean Water Act.

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

Parameter[1]	Method	EPA[2 7]	Standard methods	ASTM	Other
1. Acenaphthene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
2. Acenaphthylene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
3. Acrolein	GC	603.			
	GC/MS	624,[4] 1624B.			
4. Acrylonitrile	GC	603.			
	GC/MS	624,[4] 1624B.			
5. Anthracene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440B–00	D4657–92 (99).	
6. Benzene	GC	602	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
7. Benzidine	Spectrophotometric.				See footnote,[3] p.1.
	GC/MS	625,[5] 1625B	6410 B–00.		
	HPLC	605.			
8. Benzo(a)anthracene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
9. Benzo(a)pyrene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
10. Benzo(b)fluoranthene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
11. Benzo(g,h,i)perylene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
12. Benzo(k)fluoranthene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
13. Benzylchloride	GC				See footnote,[3] p. 130.

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

Parameter[1]	Method	EPA[2][7]	Standard methods	ASTM	Other
	GC/MS				See footnote,[6] p. S102.
14. Butyl benzyl phthalate	GC	606.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
15. Bis(2-chloroethoxy) methane	GC	611.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
16. Bis(2-chloroethyl) ether	GC	611.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
17. Bis(2-ethylhexyl) phthalate	GC	606.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
18. Bromodichloromethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
19. Bromoform	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
20. Bromomethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
21. 4-Bromophenyl phenyl ether	GC	611.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
22. Carbon tetrachloride	GC	601	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
23. 4-Chloro-3-methylphenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
24. Chlorobenzene	GC	601, 602	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
25. Chloroethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
26. 2-Chloroethylvinyl ether	GC	601.			
	GC/MS	624, 1624B.			
27. Chloroform	GC	601	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
28. Chloromethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
29. 2-Chloronaphthalene	GC	612	6410 B–00.		
	GC/MS	625, 1625B			See footnote,[9] p. 27.
30. 2-Chlorophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
31. 4-Chlorophenyl phenyl ether	GC	611.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
32. Chrysene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
33. Dibenzo(a,h)anthracene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
34. Dibromochloromethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
35. 1,2-Dichlorobenzene	GC	601,602	6200 C–97.		
	GC/MS	624, 1625B	6200 B–97		See footnote,[9] p. 27.
36. 1,3-Dichlorobenzene	GC	601, 602	6200 C–97.		
	GC/MS	624, 1625B	6200 B–97		See footnote,[9] p. 27.
37. 1,4-Dichlorobenzene	GC	601, 602	6200 C–97.		
	GC/MS	624, 1625B	6200 B–97		See footnote,[9] p. 27.
38. 3,3-Dichlorobenzidine	GC/MS	625, 1625B	6410 B–00.		
	HPLC	605.			

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

Parameter[1]	Method	EPA[2,7]	Standard methods	ASTM	Other
39. Dichlorodifluoromethane	GC	601.			
	GC/MS		6200 C–97.		
40. 1,1-Dichloroethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
41. 1,2-Dichloroethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
42. 1,1-Dichloroethene	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
43. trans-1,2-Dichloroethene	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
44. 2,4-Dichlorophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
45. 1,2-Dichloropropane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
46. cis-1,3-Dichloropropene	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
47. trans-1,3-Dichloropropene	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
48. Diethyl phthalate	GC	606.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
49. 2,4-Dimethylphenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
50. Dimethyl phthalate	GC	606.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
51. Di-n-butyl phthalate	GC	606.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
52. Di-n-octyl phthalate	GC	606.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
53. 2,3-Dinitrophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00.		
54. 2,4-Dinitrotoluene	GC	609.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
55. 2,6-Dinitrotoluene	GC	609.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
56. Epichlorohydrin	GC				See footnote,[3] p. 130.
	GC/MS				See footnote,[6] p. S102.
57. Ethylbenzene	GC	602	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
58. Fluoranthene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
59. Fluorene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran	GC/MS	1613B.			
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran	GC/MS	1613B.			
62. 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	GC/MS	1613B.			
63. Hexachlorobenzene	GC	612.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
64. Hexachlorobutadiene	GC	612.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
65. Hexachlorocyclopentadiene	GC	612.			
	GC/MS	625,[5] 1625B	6410 B–00		See footnote,[9] p. 27.
66. 1,2,3,4,7,8-Hexachlorodibenzofuran	GC/MS	1613B.			
67. 1,2,3,6,7,8-Hexachlorodibenzofuran	GC/MS	1613B.			
68. 1,2,3,7,8,9-Hexachlorodibenzofuran	GC/MS	1613B.			

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

Parameter[1]	Method	EPA[2][7]	Standard methods	ASTM	Other
69. 2,3,4,6,7,8-Hexachlorodibenzofuran	GC/MS	1613B.			
70. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	GC/MS	1613B.			
71. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	GC/MS	1613B.			
72. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	GC/MS	1613B.			
73. Hexachloroethane	GC	612.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
74. Ideno(1,2,3-cd) pyrene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
75. Isophorone	GC	609.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
76. Methylene chloride	GC	601	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
77. 2-Methyl-4,6-dinitrophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
78. Naphthalene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00.		
79. Nitrobenzene	GC	609.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC			D4657–92 (99).	
80. 2-Nitrophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
81. 4-Nitrophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
82. N-Nitrosodimethylamine	GC	607.			
	GC/MS	625[5], 1625B	6410 B–00		See footnote,[9] p. 27.
83. N-Nitrosodi-n-propylamine	GC	607.			
	GC/MS	625[5], 1625B	6410 B–00		See footnote,[9] p. 27.
84. N-Nitrosodiphenylamine	GC	607.			
	GC/MS	625[5], 1625B	6410 B–00		See footnote,[9] p. 27.
85. Octachlorodibenzofuran	GC/MS	1613B[10].			
86. Octachlorodibenzo-p-dioxin	GC/MS	1613B[10].			
87. 2,2′-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether].	GC	611.			
	GC/MS	625, 1625B	6410 B–00.		
88. PCB–1016	GC	608			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625	6410 B–00.		
89. PCB–1221	GC	608			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625	6410 B–00.		
90. PCB–1232	GC	608			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625	6410 B–00.		
91. PCB–1242	GC	608			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625	6410 B–00.		
92. PCB–1248	GC	608.			
	GC/MS	625	6410 B–00.		
93. PCB–1254	GC	608			See footnote,[3] p. 43; See footnote.[8]
	GC/MS	625	6410 B–00.		

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

Parameter[1]	Method	EPA[2] [7]	Standard methods	ASTM	Other
94. PCB–1260	GC	608			See footnote,[3] p. 43; See footnote.[8].
	GC/MS	625	6410 B–00.		
95. 1,2,3,7,8-Pentachloro-dibenzofuran	GC/MS	1613B.			
96. 2,3,4,7,8-Pentachloro-dibenzofuran	GC/MS	1613B.			
97. 1,2,3,7,8,-Pentachlorodibenzo-p-dioxin	GC/MS	1613B.			
98. Pentachlorophenol	GC	604	6420 B–00		See footnote,[3] p. 140.
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
99. Phenanthrene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
100. Phenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
101. Pyrene	GC	610.			
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
	HPLC	610	6440 B–00	D4657–92 (99).	
102. 2,3,7,8-Tetra-chlorodibenzofuran	GC/MS	1613B.			
103. 2,3,7,8-Tetra-chlorodibenzo-p-dioxin	GC/MS	613, 625,[5a] 1613B.			
104. 1,1,2,2-Tetra-chloro ethane	GC	601	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
105. Tetrachloroethene	GC	601	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
106. Toluene	GC	602	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
107. 1,2,4-Trichlorobenzene	GC	612			See footnote,[3] p. 130.
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
108. 1,1,1-Trichloroethane	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
109. 1,1,2-Trichloroethane	GC	601	6200 C–97		See footnote,[3] p. 130.
	GC/MS	624, 1624B	6200 B–97.		
110. Trichloroethene	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
111. Trichlorofluoromethane	GC	601	6200 C–97.		
	GC/MS	624	6200 B–97.		
112. 2,4,6-Trichlorophenol	GC	604	6420 B–00.		
	GC/MS	625, 1625B	6410 B–00		See footnote,[9] p. 27.
113. Vinyl chloride	GC	601	6200 C–97.		
	GC/MS	624, 1624B	6200 B–97.		
114. Nonylphenol	GC/MS			D7065–06.	
	LC/MS/MS			D7485–09.	
115. Bisphenol A (BPA)	GC/MS			D7065–06.	
	LC/MS/MS			D7574–09.	
116. p-tert-Octylphenol (OP)	GC/MS			D7065–06.	
	LC/MS/MS			D7485–09.	
117. Nonylphenol Monoethoxylate (NP1EO)	GC/MS			D7065–06.	
	LC/MS/MS			D7485–09.	
118. Nonylphenol Diethoxylate (NP2EO)	GC/MS			D7065–06.	
	LC/MS/MS			D7485–09.	
119. Polybrominated diphenyl ethers (PBDEs) 49 congeners.	HRGC/HRMS	1614A.			
120. Polychlorinated biphenyls (PCBs) 209 Congeners.	HRGC/HRMS	1668C.			
121. Adsorbable Organic Halides (AOX)	Adsorption and Coulometric Titration.	1650.			

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

Parameter[1]	Method	EPA[2][7]	Standard methods	ASTM	Other
122. Chlorinated Phenolics	In Situ Acetylation and GC/MS.	1653.			

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, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The full text of Method 1613B is incorporated by reference into this Part 136 and is available from the National Technical Information Services as stock number PB95–104774. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136. The full text of Methods 1613B, 1614A, 1650, 1653, and 1668C are available from EPA Office of Water (4303T) 1200 Pennsylvania Ave, NW, Washington, DC 20460.

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

[4] Method 624 may be used for definitive determination of Acrolein and Acrylonitrile.

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

[5a] 625, screening only.

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

[7] Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624B, and 1625B (See Appendix A of this Part 136) in accordance with procedures each in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624B and 1625B) of all samples to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

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

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

[10] Analysts may use Fluid Management Systems, Inc. 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.

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

Parameter	Method	EPA[2][7][10]	Standard methods	ASTM	Other
1. Aldrin	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96 (02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		
2. Ametryn	GC	507, 619	....	....	See footnote,[3] p. 83; See footnote,[9] O–3106–93; See footnote,[6] p. S68.
	GC/MS	525.1, 525.2	....	....	See footnote,[14] O–1121–91.
3. Aminocarb	TLC	....	....	....	See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
4. Atraton	GC	619	....	....	See footnote,[3] p. 83; See footnote,[6] p. S68.
5. Atrazine	GC	507, 619	....	....	See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	HPLC/MS	....	....	....	See footnote,[12] O–2060–01.
	GC/MS	525.1, 525.2	....	....	See footnote,[11] O–1126–95.
6. Azinphos methyl	GC	614, 622, 1657	....	....	See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC MS	....	....	....	See footnote,[11] O–1126–95.
7. Barban	TLC	....	....	....	See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
8. α-BHC	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[8] 3M0222.
	GC/MS	625[5]	6410 B–00	....	See footnote,[11] O–1126–95.
9. β-BHC	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		
10. δ-BHC	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[8] 3M0222.

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

Parameter	Method	EPA[2,7,10]	Standard methods	ASTM	Other
	GC/MS	625	6410 B–00.		
11. γ-BHC (Lindane)	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625[5]	6410 B–00		See footnote,[11] O–1126–95.
12. Captan	GC	617	6630 B–00	D3086–90, D5812–96(02).	See footnote,[3] p. 7.
13. Carbaryl	TLC				See footnote,[3] p. 94, See footnote,[6] p. S60.
	HPLC	531.1, 632.			
	HPLC/MS	553			See footnote,[12] O–2060–01.
	GC/MS				See footnote,[11] O–1126–95.
14. Carbophenothion	GC	617	6630 B–00		See footnote,[6] p. S73.
15. Chlordane	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		
16. Chloropropham	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
17. 2,4-D	GC	615	6640 B–01		See footnote,[3] p. 115; See footnote,[4] O–3105–83.
	HPLC/MS				See footnote,[12] O–2060–01.
18. 4,4′-DDD	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3105–83; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		
19. 4,4′-DDE	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		See footnote,[11] O–1126–95.
20. 4,4′–DDT	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		
21. Demeton-O	GC	614, 622			See footnote,[3] p. 25; See footnote,[6] p. S51.
22. Demeton-S	GC	614, 622			See footnote,[3] p. 25; See footnote,[6] p. S51.
23. Diazinon	GC	507, 614, 622, 1657			See footnote,[3] p. 25; See footnote,[4] O–3104–83; See footnote,[6] p. S51.
	GC/MS	525.1, 525.2			See footnote,[11] O–1126–95.
24. Dicamba	GC	615			See footnote,[3] p. 115.
	HPLC/MS				See footnote,[12] O–2060–01.
25. Dichlofenthion	GC	622.1			See footnote,[6] p. S73.
26. Dichloran	GC	608.2, 617	6630 B–00		See footnote,[3] p. 7.
27. Dicofol	GC	617	6630 B–00		See footnote,[4] O–3104–83.
28. Dieldrin	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00		See footnote,[11] O–1126–95.
29. Dioxathion	GC	614.1, 1657			See footnote,[6] p. S73.
30. Disulfoton	GC	507, 614, 622, 1657			See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC/MS	525.1, 525.2			See footnote,[11] O–1126–95.
31. Diuron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS	553			See footnote,[12] O–2060–01.
32. Endosulfan I	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M022.
	GC/MS	625[5]	6410 B–00		See footnote,[13] O–2002–01.
33. Endosulfan II	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[8] 3M0222.
	GC/MS	625[5]	6410 B–00		See footnote,[13] O–2002–01.
34. Endosulfan Sulfate	GC	608, 617	6630 C–00		See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		

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

Parameter	Method	EPA [2] [7] [10]	Standard methods	ASTM	Other
35. Endrin	GC	505, 508, 608, 617, 1656	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	525.1, 525.2, 625 [5]	6410 B–00.		
36. Endrin aldehyde	GC	608, 617	6630 C–00		See footnote,[8] 3M0222.
	GC/MS	625.			
37. Ethion	GC	614, 614.1,1657			See footnote,[6] p. S73.
	GC/MS				See footnote,[13] O–2002–01.
38. Fenuron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
39. Fenuron-TCA	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
40. Heptachlor	GC	505, 508, 608, 617, 1656	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	525.1, 525.2, 625	6410 B–00.		
41. Heptachlor epoxide	GC	608, 617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[6] p. S73; See footnote,[8] 3M0222.
	GC/MS	625	6410 B–00.		
42. Isodrin	GC	617	6630 B–00 & C–00.		See footnote,[4] O–3104–83; See footnote,[6] p. S73.
43. Linuron	GC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS	553			See footnote,[12] O–2060–01.
	GC/MS				See footnote,[11] O–1126–95.
44. Malathion	GC	614, 1657	6630 B–00		See footnote,[3] p. 25; See footnote,[6] p. S51.
	GC/MS				See footnote,[11] O–1126–95.
45. Methiocarb	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
46. Methoxychlor	GC	505, 508, 608.2, 617, 1656.	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83; See footnote,[8] 3M0222.
	GC/MS	525.1, 525.2			See footnote,[11] O–1126–95.
47. Mexacarbate	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
48. Mirex	GC	617	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 7; See footnote,[4] O–3104–83.
49. Monuron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
50. Monuron-TCA	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
51. Neburon	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
52. Parathion methyl	GC	614, 622, 1657	6630 B–00		See footnote,[3] p. 25.
	GC/MS				See footnote,[11] O–1126–95.
53. Parathion ethyl	GC	614	6630 B–00		See footnote,[3] p. 25.
	GC/MS				See footnote,[11] O–1126–95.
54. PCNB	GC	608.1, 617	6630 B–00	D3086–90, D5812–96(02).	See footnote,[3] p. 7.
55. Perthane	GC	617	6630 B–00	D3086–90, D5812–96(02).	See footnote,[4] O–3104–83.
56. Prometon	GC	507, 619			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2			See footnote,[11] O–1126–95.

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

Parameter	Method	EPA[2][7][10]	Standard methods	ASTM	Other
57. Prometryn	GC	507, 619			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2			See footnote,[13] O–2002–01.
58. Propazine	GC	507, 619, 1656			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2.			
59. Propham	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
60. Propoxur	TLC				See footnote,[3] p. 94; See footnote,[6] p. S60.
	HPLC	632.			
61. Secbumeton	TLC				See footnote,[3] p. 83; See footnote,[6] p. S68.
	GC	619.			
62. Siduron	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
	HPLC/MS				See footnote,[12] O–2060–01.
63. Simazine	GC	505, 507, 619, 1656			See footnote,[3] p. 83; See footnote,[6] p. S68; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2			See footnote,[11] O–1126–95.
64. Strobane	GC	617	6630 B–00 & C–00.		See footnote,[3] p. 7.
65. Swep	TLC				See footnote,[3] p. 104; See footnote,[6] p. S64.
	HPLC	632.			
66. 2,4,5-T	GC	615	6640 B–01		See footnote,[3] p. 115; See footnote,[4] O–3105–83.
67. 2,4,5-TP (Silvex)	GC	615	6640 B–01		See footnote,[3] p. 115; See footnote,[4] O–3105–83.
68. Terbuthylazine	GC	619, 1656			See footnote,[3] p. 83; See footnote,[6] p. S68.
	GC/MS				See footnote,[13] O–2002–01.
69. Toxaphene	GC	505, 508, 608, 617, 1656	6630 B–00 & C–00.	D3086–90, D5812–96(02).	See footnote,[3] p. 115; See footnote,[4] O–3105–83.
	GC/MS	525.1, 525.2, 625	6410 B–00.		
70. Trifluralin	GC	508, 617, 627, 1656	6630 B–00		See footnote,[3] p. 7; See footnote,[9] O–3106–93.
	GC/MS	525.1, 525.2			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, where entries are listed by chemical name.

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

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

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

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

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

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

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

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

[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.

[11] O–1126–95 GC/MS: Zaugg, S.D., Sandstrom, M.W., Smith, S.G., and Fehlberg, K.M., 1995, 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: U.S. Geological Survey Open-File Report 95–181, Method O–1126–95, 49 p.

[12] O–2060–01 LC/MS: Furlong, E.T., Anderson, B.D., Werner, S.L., Soliven, P.P., Coffey, L.J., and Burkhardt, M.R., 2001, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory-Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry: U.S. Geological Survey Water-Resources Investigations Report 01–4134 Method O–2060–01, 73 p.

[13] O–2002–01 Sandstrom, M.W., Stroppel, M.E., Foreman, W.T., and Schroeder, M.P., 2001, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of moderate-use pesticides in water by C–18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry: U.S. Geological Survey Water-Resources Investigations Report 01–4098, Method O–2002–01, 70 p.

[14] O–1121–91: Sandstrom, M.W., Wydoski, D.S., Schroeder, M.P., Zamboni, J.L., and Foreman, W.T., 1992, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of organonitrogen herbicides in water by solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring,: U.S. Geological Survey Open-File Report 91–519; O–1121–91, 34 p.

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TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS

[40 CFR 455]

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s)
8	Triadimefon	43121–43–3	507/633/525.1/525.2/1656
12	Dichlorvos	62–73–7	1657/507/622/525.1/525.2
16	2,4-D; 2,4-D Salts and Esters [2,4-Dichloro-phenoxy-acetic acid].	94–75–7	1658/515.1/615/515.2/555
17	2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid].	94–82–6	1658/515.1/615/515.2/555
22	Mevinphos	7786–34–7	1657/507/622/525.1/525.2
25	Cyanazine	21725–46–2	629/507
26	Propachlor	1918–16–7	1656/508/608.1/525.1/525.2
27	MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid].	94–74–6	1658/615/555
30	Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid].	120–36–5	1658/515.1/615/515.2/555
31	MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid].	93–65–2	1658/615/555
35	TCMTB [2-(Thiocyanomethylthio) benzo-thiazole]	21564–17–0	637
39	Pronamide	23950–58–5	525.1/525.2/507/633.1
41	Propanil	709–98–8	632.1/1656
45	Metribuzin	21087–64–9	507/633/525.1/525.2/1656
52	Acephate	30560–19–1	1656/1657
53	Acifluorfen	50594–66–6	515.1/515.2/555
54	Alachlor	15972–60–8	505/507/645/525.1/525.2/1656
55	Aldicarb	116–06–3	531.1
58	Ametryn	834–12–8	507/619/525.1/525.2
60	Atrazine	1912–24–9	505/507/619/525.1/525.2/1656
62	Benomyl	17804–35–2	631
68	Bromacil; Bromacil Salts and Esters	314–40–9	507/633/525.1/525.2/1656
69	Bromoxynil	1689–84–5	1625/1661
69	Bromoxynil octanoate	1689–99–2	1656
70	Butachlor	23184–66–9	507/645/525.1/525.2/1656
73	Captafol	2425–06–1	1656
75	Carbaryl [Sevin]	63–25–2	531.1/632/553
76	Carbofuran	1563–66–2	531.1/632
80	Chloroneb	2675–77–6	1656/508/608.1/525.1/525.2
82	Chlorothalonil	1897–45–6	508/608.2/525.1/525.2/1656
84	Stirofos	961–11–5	1657/507/622/525.1/525.2
86	Chlorpyrifos	2921–88–2	1657/508/622
90	Fenvalerate	51630–58–1	1660
103	Diazinon	333–41–5	1657/507/614/622/525.1/525.2
107	Parathion methyl	298–00–0	1657/614/622
110	DCPA [Dimethyl 2,3,5,-tetrachloro-terephthalate]	1861–32–1	508/608.2/525.1/525.2/515.1/515.2/1656
112	Dinoseb	88–85–7	1658/515.1/615/515.2/555
113	Dioxathion	78–34–2	1657/614.1
118	Nabonate [Disodium cyanodithio-imidocarbonate]	138–93–2	630.1
119	Diuron	330–54–1	632/553
123	Endothall	145–73–3	548/548.1
124	Endrin	72–20–8	1656/505/508/608/617/525.1/525.2
125	Ethalfluralin	55283–68–6	1656/627 Note 1
126	Ethion	563–12–2	1657/614/614.1
127	Ethoprop	13194–48–4	1657/507/622/525.1/525.2
132	Fenarimol	60168–88–9	507/633.1/525.1/525.2/1656
133	Fenthion	55–38–9	1657/622
138	Glyphosate [N-(Phosphonomethyl) glycine]	1071–83–6	547

TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS—Continued

[40 CFR 455]

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s)
140	Heptachlor	76–44–8	1656/505/508/608/617/525.1/525.2
144	Isopropalin	33820–53–0	1656/627
148	Linuron	330–55–2	553/632
150	Malathion	121–75–5	1657/614
154	Methamidophos	10265–92–6	1657
156	Methomyl	16752–77–5	531.1/632
158	Methoxychlor	72–43–5	1656/505/508/608.2/617/525.1/525.2
172	Nabam	142–59–6	630/630.1
173	Naled	300–76–5	1657/622
175	Norflurazon	27314–13–2	507/645/525.1/525.2/1656
178	Benfluralin	1861–40–1	[1]1656/[1]627
182	Fensulfothion	115–90–2	1657/622
183	Disulfoton	298–04–4	1657/507/614/622/525.1/525.2
185	Phosmet	732–11–6	1657/622.1
186	Azinphos Methyl	86–50–0	1657/614/622
192	Organo-tin pesticides	12379–54–3	Ind-01/200.7/200.9
197	Bolstar	35400–43–2	1657/622
203	Parathion	56–38–2	1657/614
204	Pendimethalin	40487–42–1	1656
205	Pentachloronitrobenzene	82–68–8	1656/608.1/617
206	Pentachlorophenol	87–86–5	625/1625/515.2/555/515.1/525.1/525.2
208	Permethrin	52645–53–1	608.2/508/525.1/525.2/1656/1660
212	Phorate	298–02–2	1657/622
218	Busan 85 [Potassium dimethyldithiocarbamate]	128–03–0	630/630.1
219	Busan 40 [Potassium N-hydroxymethyl-N-methyldithiocarbamate].	51026–28–9	630/630.1
220	KN Methyl [Potassium N-methyl-dithiocarbamate]	137–41–7	630/630.1
223	Prometon	1610–18–0	507/619/525.1/525.2
224	Prometryn	7287–19–6	507/619/525.1/525.2
226	Propazine	139–40–2	507/619/525.1/525.2/1656
230	Pyrethrin I	121–21–1	1660
232	Pyrethrin II	121–29–9	1660
236	DEF [S,S,S-Tributyl phosphorotrithioate]	78–48–8	1657
239	Simazine	122–34–9	505/507/619/525.1/525.2/1656
241	Carbam-S [Sodium dimethyldithiocarbanate]	128–04–1	630/630.1
243	Vapam [Sodium methyldithiocarbamate]	137–42–8	630/630.1
252	Tebuthiuron	34014–18–1	507/525.1/525.2
254	Terbacil	5902–51–2	507/633/525.1/525.2/1656
255	Terbufos	13071–79–9	1657/507/614.1/525.1/525.2
256	Terbuthylazine	5915–41–3	619/1656
257	Terbutryn	886–50–0	507/619/525.1/525.2
259	Dazomet	533–74–4	630/630.1/1659
262	Toxaphene	8001–35–2	1656/505/508/608/617/525.1/525.2
263	Merphos [Tributyl phosphorotrithioate]	150–50–5	1657/507/525.1/525.2/622
264	Trifluralin	1582–09–8	1656/508/617/627/525.1/525.2
268	Ziram [Zinc dimethyldithiocarbamate]	137–30–4	630/630.1

[1] Monitor and report as total Trifluralin.

TABLE IH—LIST OF APPROVED MICROBIOLOGICAL METHODS FOR AMBIENT WATER

Parameter and units	Method[1]	EPA	Standard methods	AOAC, ASTM, USGS	Other
Bacteria:					
1. Coliform (fecal), number per 100 mL or number per gram dry weight.	Most Probable Number (MPN), 5 tube, 3 dilution, or.	p. 132[3]	9221 C E–2006.		
	Membrane filter (MF)[2], single step.	p. 124[3]	9222 D–1997	B–0050–85.[4]	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 132[3]	9221 C E–2006.		
	MF[2], single step	p. 124[3]	9222 D–1997.		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 114[3]	9221 B–2006.		
	MF[2], single step or two step.	p. 108[3]	9222 B–1997	B–0025–85.[4]	

TABLE IH—LIST OF APPROVED MICROBIOLOGICAL METHODS FOR AMBIENT WATER—Continued

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

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

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

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

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

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

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

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

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

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

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

[11] The multiple-tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

[12] These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by *E. coli.*

[13] After prior enrichment in a presumptive medium for total coliform using 9221B.1, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

[14] Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray® or Quanti-Tray®/2000, and the MPN calculated from the table provided by the manufacturer.

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

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

[17] A description of the mColiBlue24® test may be obtained from Hach Company, 100 Dayton Ave., Ames, IA 50010.

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

[19] USEPA. March 2010. Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–10–002.

[20] USEPA. December 2009. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–09–007.

[21] Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. "New Medium for the Simultaneous Detection of Total Coliform and *Escherichia coli* in Water." Appl. Environ. Microbiol. 59:3534–3544 and in USEPA. September 2002.: Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA 821–R–02–024.

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

[23] USEPA. December 2009. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE–EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–09–015.

[24] USEPA. December 2009. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β–D–Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–09–016.

[25] Method 1622 uses a filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium.* USEPA. December 2005. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–05–001.

[26] Method 1623 uses a filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. USEPA. December 2005. Method 1623. *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–05–002.

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(b) * * *

(1) The full texts of the CWA U.S. EPA methods are available at *http://epa.gov/waterscience/methods/method.* The full text for determining the method detection limit when using the test procedures is given in appendix B of this part 136.

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(54) USEPA. March 2010. Method 1103.1: *Escherichia coli (E. coli)* in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–621–R–10–002. Available at *http://www.epa.gov/waterscience/methods/method.* Table IH, Note 19.

(55) USEPA. December 2009. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE–EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–621–R–09–015. Available at *http://www.epa.gov/waterscience/methods/method.* Table IH, Note 23.

(56) USEPA. December 2009. Method 1603: *Escherichia coli (E. coli)* in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–09–007. Available at *http://www.epa.gov/waterscience/methods/method.* Table IA, Note 20; Table IH, Note 20.

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(59) USEPA. December 2009. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–09–016. Available at *http://www.epa.gov/waterscience/methods/method.* Table IA, Note 23; Table IH, Note 24.

(60) USEPA. December 2005. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–05–001. Available at *http://www.epa.gov/waterscience/methods/method.* Table IA, Note 25.

(61) USEPA. December 2005. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–05–002. Available at *http://www.epa.gov/waterscience/methods/method.* Table IA, Note 26.

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(70) USEPA. April 2010. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using Lauryl Tryptose Broth (LTB) and EC Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–10–003. Available at *http://www.epa.gov/waterscience/methods/method.* Table IA, Note 13.

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(73) EPA Method 200.5, Revision 4.2. "Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry." 2003. EPA/600/R–06/115. (Available at *http://www.epa.gov/nerlcwww/ordmeth.htm.*)

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(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters are cited in Tables IA, IB, IC, ID, IE, IF, IG and IH are prescribed in Table II. Information in this table takes precedence over information provided in specific methods or elsewhere unless a party documents the acceptability of an alternative to the Table II instructions. Such alternatives may include a change from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples collected from a specific discharge. The nature and extent of the documentation of such changes (how to apply as well as supporting data) is left to the discretion of the permitting authority (state agency or EPA region) or other authority and may rely on instructions, such as those provided for method modifications at § 136.6.

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

Parameter number/name	Container [1]	Preservation [2,3]	Maximum holding time (in hours) [4]
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and *E. coli*	PA, G	Cool, < 10 °C, 0.008% $Na_2S_2O_3$ [5].	[22,23] 8
6. Fecal streptococci	PA, G	Cool, < 10 °C, 0.008% $Na_2S_2O_3$ [5].	[22] 8
7. Enterococci	PA, G	Cool, < 10 °C, 0.008% $Na_2S_2O_3$ [5].	[22] 8
8. *Salmonella*	PA, G	Cool, < 10 °C, 0.008% $Na_2S_2O_3$ [5].	[22] 8
Table IA—Aquatic Toxicity Tests: 9–12. Toxicity, acute and chronic.	P, FP, G	Cool, 0–6 °C [16]	36
* * *	* *	*	*
Table IH—Bacterial Tests:			

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

Parameter number/name	Container[1]	Preservation[2,3]	Maximum holding time (in hours)[4]
1. *E. coli*	PA, G	Cool, < 10 °C, 0.008% $Na_2S_2O_3$[5].	[22]8
2. Enterococci	PA, G	Cool, < 10 °C, 0.008% $Na_2S_2O_3$[5].	[22]8
Table IH—Protozoan Tests:			
8. *Cryptosporidium*	LDPE; field filtration	1–10 °C	[21]96
9. *Giardia*	LDPE; field filtration	1–10 °C	[21]96

[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.

[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°, 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 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

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

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

[5] ASTM D7365–09a specifies treatment options for samples containing oxidants (*e.g.* chlorine).

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

* * * * * * *

[16] Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.

[21] Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

[22] Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

[23] For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

* * * * *

4. Section 136.4 is revised to read as follows:

§ 136.4 Application for and approval of alternate test procedures for nationwide use.

(a) A written application for review of an alternate test procedure (alternate method) for nationwide use may be made by letter via email or by hard copy in triplicate to the National Alternate Test Procedure Program Coordinator (National Coordinator), Office of Science and Technology (4303T), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460. Any application for an alternate test procedure (ATP) under this paragraph shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide

approval of an alternate test procedure is being requested.

(3) Provide a detailed description of the proposed alternate test procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure for the analysis of the pollutant(s) or parameter(s) in wastewater discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternative test procedure compared to the performance of the reference method.

(b) The National Coordinator may request additional information and analyses from the applicant in order to determine whether the alternate test procedure satisfies the applicable requirements of this

(c) *Approval for nationwide use.* (1) After a review of the application and any additional analyses requested from the applicant, the National Coordinator will notify the applicant, in writing, of acceptance or rejection of the alternate test procedure for nationwide use in CWA programs. If the application is not approved, the National Coordinator will specify what additional information might lead to a reconsideration of the application, and notify the Regional Alternate Test Procedure Coordinators of such rejection. Based on the National Coordinator's rejection of a proposed alternate test procedure and an assessment of any approvals for limited uses for the unapproved method, the Regional Coordinator may decide to withdraw approval of the method for limited use in the Region.

(2) Where the National Coordinator approved an applicant's request for nationwide use of an alternate test procedure, the National Coordinator will notify the applicant that the National Coordinator will recommend rulemaking to approve the alternate test procedure. The National Coordinator will notify the Regional Coordinators that they may consider approval of this alternate test procedure for limited use in their Regions based on the information and data provided in the applicant's application.

(3) EPA will propose to amend 40 CFR part 136 to include the alternate test procedure in § 136.3. EPA shall make available for review all the factual bases for its proposal, including any performance data submitted by the applicant and any available EPA analysis of those data.

(4) Following public comment, EPA shall publish in the **Federal Register** a final decision on whether to amend 40 CFR part 136 to include the alternate test procedure as an approved analytical method.

(5) Whenever the National Coordinator has approved an applicant's request for nationwide use of an alternate test procedure, any person may request an approval of the method for limited use under § 136.5 from the EPA Region.

5. Section 136.5 is revised to read as follows:

§ 136.5 Approval of alternate test procedures for limited use.

(a) Any person may request the Regional Alternate Test Procedure Coordinator to approved the use of an alternate test procedure in the Region.

(b) When the request for the use of an alternate test procedure concerns use in a State with an NPDES permit program approved pursuant to section 402 of the Act, the requestor, shall first submit an application for limited use to the Director of the State agency having responsibility for issuance of NPDES permits within such State. The Director will forward the application to the Regional Coordinator with a recommendation for or against approval.

(c) Any application for approval of an alternate test procedure for limited use may be made by letter, email or by hard copy. The application shall include the following:

(1) Provide the name and address of the applicant and the applicable ID number of the existing or pending permit and issuing agency for which use of the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate test procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I or in the NPDES permit.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(d) Approval for limited use. (1) After a review of the application and in the case of a State with an approved NPDES permit program, review of the recommendation of the Director, the Regional Coordinator will notify the applicant and the appropriate State agency of approval or rejection of the use of the alternate procedure. The approval may be restricted to use only with request to a specific discharge or facility (and its laboratory) or, at the discretion of the Regional Coordinator, to all discharger or facilities (and their associated laboratories) specified in the approval for the Region. If the application for approval is not approved, the Regional Coordinator shall specify what additional information might lead to a reconsideration of the application.

(2) The Regional Coordinator will forward a copy of every approval and rejection notification to the National Alternate Test Procedure Coordinator.

6. Section 136.6 is revised to read as follows:

§ 136.6 Method modifications and analytical requirements.

(a) *Definitions of terms used in this section.*

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

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

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

(4) *Equivalent performance* means that the modified method produces results that meet or exceed the QC acceptance criteria of the approved method.

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

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

(b) *Method modifications.* (1) If the underlying chemistry and determinative technique in a modified method are essentially the same as an unmodified part 136 method, then the modified method is an equivalent and acceptable alternative to the approved method. However, those who develop or use a modification to an approved (part 136) method must document that the performance of the modified method, in the matrix to which the modified method will be applied, is equivalent to the performance of the approved method. This documentation should include the routine initial demonstration of capability and ongoing QC including determination of precision and accuracy, detection limits, and matrix spike recoveries. Initial demonstration of capability typically includes analysis of a four replicate mid-level standard and a method detection limit study. Ongoing quality control typically includes method blanks, mid-level laboratory control samples, and matrix spikes. The method is considered equivalent if the quality control requirements in the reference method are achieved. The method user's Standard Operating Procedure (SOP) must clearly document the modifications made to the reference method. Examples of allowed method modifications are listed below. The user must notify their permitting authority and/or their certification authority/ accreditation body of the intent to use a modified method when accreditation is requested. Such notification should be of the form "Method xxx has been modified within the flexibility allowed in 40 CFR Part 136.6". Specific details of the modification need not be provided, but must be documented in the Standard Operating Procedure (SOP). The certification authority/ accreditation body may request a copy of the SOP.

(2) *Requirements.* The modified method must have sufficient sensitivity to meet the data quality objectives. The modified method must also meet or exceed performance of the approved method(s) for the analyte(s) of interest, as documented by meeting the initial and ongoing quality control requirements in the method.

(i) *Requirements for establishing equivalent performance.* If the approved method contains QC tests and QC acceptance criteria, the modified method must use these QC tests and the modified method must meet the QC acceptance criteria with the following conditions:

(A) The analyst may only rely on QC tests and QC acceptance criteria in a method if it includes wastewater matrix QC tests and QC acceptance criteria (*e.g.,* matrix spikes) and both initial (start-up) and ongoing QC tests and QC acceptance criteria.

(B) If the approved method does not contain QC tests and QC acceptance criteria or if the QC tests and QC acceptance criteria in the method do not meet the requirements of this section, then the analyst must employ QC tests published in the "equivalent" or part 136 method that has such QC, or the essential QC requirements specified at 136.7. If the QC requirements are sufficient, but published in other parts of an organization's compendium rather than within the part 136 method then that part of the organization's compendium must be used.

(C) In addition, the analyst must perform ongoing QC tests, including assessment of performance of the modified method on the sample matrix (*e.g.,* analysis of a matrix spike/matrix spike duplicate pair for every twenty samples), and analysis of an ongoing precision and recovery sample (*e.g.,* laboratory fortified blank or blank spike) and a blank with each batch of 20 or fewer samples.

(D) Calibration must be performed using the modified method. The modified method must be tested with every wastewater matrix and be applied to up to nine distinct matrices in addition to any and all reagent water tests. If the performance in the wastewater matrix or reagent water does not meet the QC acceptance criteria, the method modification may not be used.

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

(3) *Restrictions.* An analyst may not modify an approved Clean Water Act analytical method for a method-defined analyte. In addition, an analyst may not modify an approved method if the modification would result in measurement of a different form or species of an analyte. Changes in method parameters are not allowed if such changes would alter the defined methodology (i.e. method principle) of the unmodified method. For example, phenol method 420.1 or 420.4 defines phenolics as ferric iron oxidized compounds that react with 4-aminoantipyrine (4–AAP) at pH 10 after being distilled from acid solution. Because total phenolics represents a group of compounds that all react at different efficiencies with 4–AAP, changing test conditions likely would change the behavior of these different phenolic compounds. An analyst may not modify any sample preservation and/or holding time requirements of an approved method.

(4) *Allowable changes.* Except as noted under *Restrictions* of this section, an analyst may modify an approved test procedure (analytical method) provided the underlying reactions and principles used in the approved method remain essentially the same and provided that the requirements of this section are met. If equal or better performance can be obtained with an alternative reagent, then it is allowed. These changes refer to modifications of the analytical procedures used for identification and measurement of the analyte and do not apply to sample collection and preservation procedures. Some examples of these types of changes are:

(A) Use of gas diffusion in place of manual or automated distillation.

(B) Changes in equipment operating parameters such as the monitoring wavelength of a colorimeter or the reaction time and temperature as needed to achieve the chemical reactions defined in the unmodified CWA method. For example, molybdenum blue phosphate methods have two absorbance maxima, one at about 660 nm and another at about 880 nm. The former is about 2.5 times less sensitive than the latter. Wavelength choice provides a cost effective, dilution free means to increase sensitivity of molybdenum blue phosphate methods.

(C) Interchange of oxidants, such as the use of titanium oxide in UV assisted automated digestion of TOC and total Phosphorus as long as complete oxidation can be demonstrated.

(5) *Previously Accepted Modifications.* The following modifications have been used successfully in the laboratory community for many years. Data have demonstrated that these modifications provide equivalent performance to the methods approved at part 136 across a wide variety of matrix types. Therefore, these modifications are allowed without the need to generate additional equivalency data, or the specific notification of permitting and/or certification authority/accreditation bodies required for novel method modifications. However, a laboratory wishing to use these modifications must continue to demonstrate acceptable method performance by performing and documenting all applicable initial demonstration of capability and ongoing QC tests and meeting all applicable QC acceptance criteria as described in § 136.7.

(i) Changes between manual method, flow analyzer and discrete instrumentation.

(ii) Changes in chromatographic columns or temperature programs.

(iii) Changes between automated and manual sample preparation, such as digestions, distillations, and extractions; in-line sample preparation is an acceptable form of automated sample preparation for CWA methods.

(iv) In general, ICP–MS is a sensitive and selective detector for metal analysis; however, isobaric interference can cause problems for quantitative determination as well as identification based on the isotope pattern. Interference reduction technologies, such as collision or reaction cells, are designed to reduce the effect of spectroscopic interferences that may bias results for the element of interest. The use of interference reduction technologies is allowed provided the method performance specifications relevant to ICP–MS measurements are met.

(v) The use of EPA Method 200.2 or the sample preparation steps from EPA Method 1638 including the use of closed vessel digestion is allowed for EPA Method 200.8 provided the method performance specifications relevant to the ICP–MS are met.

(vi) Changes in pH adjustment reagents. Changes in compounds used to adjust pH are acceptable as long as they do not produce interference. For example, using a different acid to adjust pH in colorimetric methods.

(vi) Changes in buffer reagents are acceptable provided that the changes do not produce interferences.

(viii) Changes in the order of reagent addition are acceptable provided that the change does not produce interference. For example using the same reagents, but adding them in different order or preparing them in combined or separate solutions (so they can be added separately), is allowed provided reagent stability or method performance is improved.

(ix) Changes in calibration range (provided that the modified range covers any relevant regulatory limit.)

(x) Changes in calibration model. Linear calibration models do not adequately fit calibration data with one or two inflection points. For example, vendor-supplied data acquisition and processing software provides quadratic fitting functions to handle such situations. If calibration data for a particular analytical method routinely display quadratic character, using quadratic fitting functions is acceptable. In such cases, the minimum number of calibrators for second order fits should be six and in no case should concentrations be extrapolated for instrument responses that exceed that of the most concentrated calibrator. Examples of methods with nonlinear calibration functions include chloride by SM4500–Cl–E–1997, hardness by EPA 130.1, cyanide by ASTM D6888 or OIA1677, Kjeldahl nitrogen by PAI–DK03, and anions by EPA 300.0. When a regression curve is calculated as an alternative to using the average response factor, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$RSE = 100x\sqrt{\frac{\sum_{i=1}^{n}\left(\frac{C_i - PC_i}{C_i}\right)^2}{n-p}}$$

n = Number of calibration points

p = Number of parameters in the model (1 for linear through the origin, 2 for linear not through the origin, 3 for quadratic, etc.)

C_i = True concentration of the standard at level i

PC_i = Predicted concentration at level i, using the calibration model chosen.

Using the RSE as a metric has the added advantage of allowing the same numerical standard to be applied to the calibration model, regardless of the form of the model. Thus, if a method states that the RSD should be ≤20% for the traditional linear model through the origin, then the RSE acceptance limit can remain ≤20% as well. Similarly, if a method provides an RSD acceptance limit of ≤15%, then that same figure can be used as the acceptance limit for the RSE. RSE may be used as an alternative to correlation coefficients and coefficients of determination for evaluating calibration curves for any of the methods at part 136. If the method includes a numerical criterion for the RSD, then the same numerical value is used for the RSE. Some older methods do not include any criterion for the calibration curve—for these methods if RSE is used the value should be ≤20%. Note that RSE is included as an alternative to correlation coefficient as a measure of the suitability of a calibration curve. It is not necessary to evaluate both RSE and correlation coefficients.

(xi) Changes in equipment such as using similar equipment from a vendor different from that mentioned in the method.

(xii) The use of micro or midi distillation apparatus in place of macro distillation apparatus.

(xiii) The use of prepackaged reagents.

(xiv) The use of digital titrators and methods where the underlying chemistry used for the determination is similar to that used in the approved method.

(xv) Use of Selected Ion Monitoring (SIM) mode for analytes that cannot be effectively analyzed in full scan mode and reach the required minimum detectible concentration. False positives are more of a concern when using SIM analysis, so at a minimum, one quantitation and two qualifying ions must be monitored for each analyte (unless less than three ions with intensity greater than 15% of the base peak are available). The ratio of the two qualifying ions to the quantitation ion must be evaluated and should agree with the ratio of an authentic standard within plus/minus 20 percent. Analyst judgment must be applied to the evaluation of ion ratios since the ratios can be affected by co-eluting matrix compounds. The signal to noise ratio of the least sensitive ion should be at least 3:1. Retention time should match within 0.05 minute of an authentic standard analyzed under identical conditions. Matrix compounds can cause minor shifts in retention time and can be evaluated by observing any shifts in the retention times of the internal standards. The total scan time should be such that a minimum of eight scans are obtained per chromatographic peak.

(xvi) Changes are allowed in purge-and-trap sample volumes or operating conditions. Some examples are:

(A) Changes in purge time and purge-gas flow rate. A change in purge time and purge-gas flow rate is allowed provided sufficient total purge volume is used to achieve the required minimum detectible concentration and calibration range for all compounds. In general, a purge rate in the range 20–200 mL/min and a total purge volume in the range 240–880 mL are recommended.

(B) Use of nitrogen or helium as a purge gas provided that the minimum detectible concentrations for all compounds are met. Using nitrogen as a purge gas can provide a significant cost saving to the laboratory, compared to helium.

(C) Sample temperature during the purge state. Gentle heating of the sample during purge (*e.g.* 40 °C) increases purge efficiency of the hydrophilic compounds and improves sample-to-sample repeatability (%RSD) because all samples are purged under precisely the same conditions.

(D) Trap sorbent. Any trap design is acceptable provided the data acquired meet all QC criteria.

(E) Changes to the desorb time. Shortening the desorb time (*e.g.* from 4 minutes to 1 minute) has no discernable effect on compound recoveries, and can shorten overall cycle time and significantly reduce the amount of water introduced to the analytical system improving the precision of analysis, especially for water soluble analytes. A desorb time of four minutes is recommended, however a desorb time in the range of 0.5–2 minutes may be used provided that all QC specifications in the method are met.

(F) Use of water management techniques is allowed. Water is always collected on the trap along with the analytes and is a significant interference for analytical systems (GC and GC/MS). Modern water management techniques (*e.g.*, dry purge or condensation points) can remove moisture from the sample stream and improve analytical performance.

(xvii) The following modifications are allowable when performing EPA Method 625: The base/neutral and acid fractions may be added together and analyzed as one extract provided that the analytes can be reliably identified and quantified in the combined extracts; the pH extraction sequence may be reversed to better separate acid and neutral components; neutral components may be extracted with either acid or base components; a smaller sample volume may be used to minimize matrix interferences provided matrix interferences are demonstrated and documented; an alternate surrogate and internal standard concentrations other than those specified in the method are acceptable provided that method performance is not degraded; an alternate calibration curve and a calibration check other than those specified in the method may be used; a different solvent for the calibration standards may be used to match the solvent of the final extract.

(xviii) If the characteristics of a wastewater matrix prevent efficient recovery of organic pollutants and prevent the method from meeting QC requirements, the analyst may attempt to resolve the issue by using salts provided that such salts do not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such salts) and that all requirements of paragraph (b)(2) of this section are met. Chlorinated samples must be dechlorinated prior to the addition of such salts.

(xix) If the characteristics of a wastewater matrix result in poor sample dispersion or reagent deposition on equipment and prevent the analyst from meeting QC requirements, the analyst may attempt to resolve the issue by adding a inert surfactant that does not affect the chemistry of the method such as Brij-35 or sodium dodecyl sulfate (SDS), provided that such surfactant does not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such surfactant) and that all requirements of paragraph (b)(1) and (b)(2) of this section are met. Chlorinated samples must be dechlorinated prior to the addition of such surfactant.

7. Add new § 136.7 to part 136 to read as follows:

§ 136.7 Quality assurance and quality control.

(a) Twelve essential Quality Control checks and acceptable abbreviations are:

(1) Demonstration of Capability (DOC);

(2) Method Detection Limit (MDL);

(3) Laboratory reagent blank (LRB), also referred to as method blank;

(4) Laboratory fortified blank (LFB), also referred to as a spiked blank, or laboratory control sample (LCS);

(5) Matrix spike, matrix spike duplicate, or laboratory fortified blank duplicate (LFBD) for suspected difficult matrices;

(6) Internal standards, surrogate standards (for organic analysis) or tracers (for radiochemistry);

(7) Calibration (initial and continuing), initial and continuing performance (ICP) solution also referred to as initial calibration verification (ICV) and continuing calibration verification (CCV);

(8) Control charts (or other trend analyses of quality control results);

(9) Corrective action (root cause analyses);

(10) QC acceptance criteria;

(11) Definitions of a batch (preparation and analytical); and

(12) Specify a minimum frequency for conducting these QC checks.

(b) These twelve quality control checks must be clearly documented in the written method along with a performance specification or description for each of the twelve quality control checks.

Appendix A [Removed and Reserved]

8. Remove and reserve Appendix A to Part 136.

Appendix C [Removed and Reserved]

9. Remove and reserve Appendix C to Part 136.

10. Revise Appendix D to Part 136 to read as follows:

Appendix D to Part 136—Precision and Recovery Statement for Methods for Measuring Metals

Two selected methods from "Methods for Chemical Analysis of Water and Wastes", EPA–600/4–79–020 (1979) have been subjected to interlaboratory method validation studies. The two selected methods are Thallium and Zinc. The following precision and recovery statements are presented in this appendix and incorporated into part 136:

Method 279.2

For Thallium, Method 279.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in "EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques)," National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 10.00–252 µg/L

$X = 0.8781(C) - 0.715$

$S = 0.1112(X) + 0.669$

$SR = 0.1005(X) + 0.241$

Where:

C = True Value for the Concentration, µg/L
X = Mean Recovery, µg/L
S = Multi-laboratory Standard Deviation, µg/L
SR = Single-analyst Standard Deviation, µg/L

Method 289.2

For Zinc, Method 289.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in "EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques)," National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.51–189 µg/L

$X = 1.6710(C) + 1.485$

$S = 0.6740(X) - 0.342$

$SR = 0.3895(X) - 0.384$

Where:

C = True Value for the Concentration, µg/L
X = Mean Recovery, µg/L
S = Multi-laboratory Standard Deviation, µg/L
SR = Single-analyst Standard Deviation, µg/L

PART 260—HAZARDOUS WASTE MANAGEMENT SYSTEM: GENERAL

11. The authority citation for part 260 continues to read as follows:

Authority: 42 U.S.C. 6905, 6912(a), 6921–6927, 6930, 6934, 6935, 6937, 6938, 6939, and 6974.

Subpart B—Definitions

12. Section 260.11 is amended by revising paragraph (c)(2) to read as follows:

§ 260.11 References.

* * * * *

(c) * * *

(2) Method 1664, Revision A and Revision 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, PB99–121949 and EPA–821–R–10–001, February 2010. IBR approved for part 261, appendix IX.

* * * * *

PART 423—STEAM ELECTRIC POWER GENERATING POINT SOURCE CATEGORY

13. The authority citation for part 423 continues to read as follows:

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

14. Section 423.11 is amended by revising paragraphs (a) and (l) to read as follows:

§ 423.11 Specialized definitions.

* * * * *

(a) The term *total residual chlorine* (or total residual oxidants for intake water with bromides) means the value obtained using any of the "chlorine-total residual" methods in Table IB 136.3(a), or other methods approved by the permitting authority.

* * * * *

(l) The term *free available chlorine* means the value obtained using any of the "chlorine-free available" methods in Table IB 136.3(a) where the method has the capability of measuring free available chlorine, or other methods approved by the permitting authority.

* * * * *

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

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

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

General Provisions

16. Section 430.01 is amended by revising paragraph (a) and by adding paragraphs (s) through (v) to read as follows:

§ 430.01 General definitions.

* * * * *

(a) *Adsorbable organic halides (AOX).* A bulk parameter that measures the total mass of chlorinated organic matter in water and wastewater. The approved method of analysis for AOX is Method 1650, listed in Table 1C at 40 CFR 136.3.

* * * * *

(s) *TCDD.* 2,3,7,8-tetrachlorodibenzop-dioxin. The approved method of analysis for TCDD is Method 1613B, listed in Table 1C at 40 CFR 136.3.

(t) *TCDF.* 2,3,7,8-tetrachlorodibenzop-furan. The approved method of analysis for TCDF is Method 1613B, listed in Table 1C at 40 CFR 136.3.

(u) Chloroform is listed with approved methods of analysis in Table 1C at 40 CFR 136.3.

(v) The approved method of analysis for the following chlorinated phenolic compounds is Method 1653, listed in Table 1C at 40 CFR 136.3:

(1) Trichlorosyringol.
(2) 3,4,5-trichlorocatechol.
(3) 3,4,6-trichlorocatechol.
(4) 3,4,5-trichloroguaiacol.
(5) 3,4,6-trichloroguaiacol.
(6) 4,5,6-trichloroguaiacol.
(7) 2,4,5-trichlorophenol.
(8) 2,4,6-trichlorophenol.
(9) Tetrachlorocatechol.
(10) Tetrachloroguaiacol.
(11) 2,3,4,6-tetrachlorophenol.
(12) Pentachlorophenol.

PART 435—OIL AND GAS EXTRACTION POINT SOURCE CATEGORY

17. The authority citation for part 435 continues to read as follows:

Authority: 33 U.S.C. 1311, 1314, 1316, 1317, 1318, 1342, and 1361.

18. Section 435.11 is amended as follows:

a. By revising paragraph (d).
b. By revising paragraph (e).
c. By revising paragraph (k)(2).
d. By revising paragraph (o).
e. By revising paragraph (t).
f. By revising paragraph (u).
g. By revising paragraph (x).
h. By revising paragraph (ee).
i. By revising paragraph (gg).
j. By revising paragraph (hh).
k. By revising paragraph (ss).
l. By adding paragraph (uu).

§ 435.11 Specialized definitions.

* * * * *

(d) *Base fluid retained on cuttings* as applied to BAT effluent limitations and NSPS refers to the "Determination of the Amount of Non-Aqueous Drilling Fluid (NAF) Base Fluid from Drill Cuttings by a Retort Chamber (Derived from API Recommended Practice 13B–2)", EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

(e) *Biodegradation rate* as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the "Protocol for the Determination of Degradation of Non Aqueous Base Fluids in a Marine Closed Bottle Biodegradation Test System: Modified ISO 11734:1995," EPA Method 1647, supplemented with "Procedure for Mixing Base Fluids With Sediments," EPA Method 1646. Both EPA Method 1646 and 1647 are published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

* * * * *

(k) * * *

(2) *Dry drill cuttings* means the residue remaining in the retort vessel after completing the retort procedure specified in EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

* * * * *

(o) *Formation oil* means the oil from a producing formation which is detected in the drilling fluid, as determined by the GC/MS compliance assurance method when the drilling fluid is analyzed before being shipped offshore, and as determined by the RPE method, EPA Method 1670, when the drilling fluid is analyzed at the offshore point of discharge. The GC/MS compliance assurance method and the RPE method approved for use with this part are published in the "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section. Detection of formation oil by the RPE method may be confirmed by the GC/MS compliance assurance method, and the results of the GC/MS compliance assurance method shall apply instead of those of the RPE method.

* * * * *

(t) *Maximum weighted mass ratio averaged over all NAF well sections* for BAT effluent limitations and NSPS for base fluid retained on cuttings means the weighted average base fluid retention for all NAF well sections as determined by EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

(u) *Method 1654A* refers to EPA Method 1654, Revision A, entitled "PAH Content of Oil by HPLC/UV," December 1992, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

* * * * *

(x) *No discharge of free oil* means that waste streams may not be discharged that contain free oil as evidenced by the monitoring method specified for that particular stream, *e.g.,* deck drainage or miscellaneous discharges cannot be discharged when they would cause a film or sheen upon or discoloration of the surface of the receiving water; drilling fluids or cuttings may not be discharged when they fail EPA Method 1617 (Static Sheen Test), which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

* * * * *

(ee) *Sediment toxicity* as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the ASTM E 1367–92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA, 19428. Copies may be inspected at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.* A copy may also be inspected at EPA's Water Docket, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

* * * * *

(gg) *SPP toxicity* as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the bioassay test procedure, "Suspended Particulate Phase (SPP) Toxicity Test," presented in EPA Method 1619, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

(hh) *Static sheen test* means the standard test procedure that has been developed for this industrial subcategory for the purpose of demonstrating compliance with the requirement of no discharge of free oil. The methodology for performing the static sheen test is presented in EPA Method 1617, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (uu) of this section.

* * * * *

(ss) *C_{16}–C_{18} internal olefin drilling fluid* means a C_{16}–C_{18} internal olefin drilling fluid formulated as specified in Appendix 1 of Subpart A of this part.

* * * * *

(uu) *Analytic Methods for the Oil and Gas Extraction Point Source Category* is the EPA document, EPA–821–R–09–013, that compiles analytic methods for this category. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be inspected at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.* A copy may also be inspected at EPA's

Water Docket, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

19. In § 435.12, the first footnote to the table is revised to read as follows:

§ 435.12 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

* * * * *

BPT EFFLUENT LIMITATIONS—OIL AND GREASE

* * * * *

* * * * *

[1] No discharge of free oil. See § 435.11(x).

* * * * *

20. In § 435.13, footnotes 2, 3, and 5 through 11 to the table are revised to read as follows:

§ 435.13 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

* * * * *

BAT EFFLUENT LIMITATIONS

* * * *

[2] As determined by the suspended particulate phase (SPP) toxicity test. See § 435.11(gg).

[3] As determined by the static sheen test. See § 435.11(hh).

* * * * *

[5] PAH mass ratio = Mass (g) of PAH (as phenanthrene)/Mass (g) of stock base fluid as determined by EPA Method 1654, Revision A, [specified at § 435.11(u)] entitled "PAH Content of Oil by HPLC/UV," December 1992, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu).

[6] Base fluid sediment toxicity ratio = 10-day LC_{50} of C_{16}–C_{18} internal olefin/10-day LC_{50} of stock base fluid as determined by ASTM E 1367–92 [specified at § 435.11(ee)] method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, after preparing the sediment according to the procedure specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu).

[7] Biodegradation rate ratio = Cumulative headspace gas production (ml) of C_{16}–C_{18} internal olefin/Cumulative headspace gas production (ml) of stock base fluid, both at 275 days as determined by EPA Method 1647, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(e) and (uu).

[8] Drilling fluid sediment toxicity ratio = 4-day LC_{50} of C_{16}–C_{18} internal olefin drilling fluid/4-day LC_{50} of drilling fluid removed from drill cuttings at the solids control equipment as determined by ASTM E 1367–92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(ee) and (uu).

[9] As determined before drilling fluids are shipped offshore by the GC/MS compliance assurance method (EPA Method 1655), and as determined prior to discharge by the RPE method (EPA Method 1670) applied to drilling fluid removed from drill cuttings. If the operator wishes to confirm the results of the RPE method (EPA Method 1670), the operator may use the GC/MS compliance assurance method (EPA Method 1655). Results from the GC/MS compliance assurance method (EPA Method 1655) shall supersede the results of the RPE method (EPA Method 1670). EPA Method 1655 and 1670 are published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu).

[10] Maximum permissible retention of non-aqueous drilling fluid (NAF) base fluid on wet drill cuttings averaged over drilling intervals using NAFs as determined by EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu). This limitation is applicable for NAF base fluids that meet the base fluid sediment toxicity ratio (Footnote 6), biodegradation rate ratio (Footnote 7), PAH, mercury, and cadmium stock limitations (C_{16}–C_{18} internal olefin) defined above in this table.

[11] Maximum permissible retention of non-aqueous drilling fluid (NAF) base fluid on wet drill cuttings averaged over drilling intervals using NAFs as determined by EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu). This limitation is applicable for NAF base fluids that meet the ester base fluid sediment toxicity ratio and ester biodegradation rate ratio stock limitations defined as:

(a) Ester base fluid sediment toxicity ratio = 10-day LC_{50} of C_{12}–C_{14} ester or C_8 ester/10-day LC_{50} of stock base fluid as determined by ASTM E 1367–92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(ee) and (uu);

(b) Ester biodegradation rate ratio = Cumulative headspace gas production (ml) of C_{12}–C_{14} ester or C_8 ester/Cumulative headspace gas production (ml) of stock base fluid, both at 275 days as determined by EPA Method 1647, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(e) and (uu); and (c) PAH mass ratio (Footnote 5), mercury, and cadmium stock limitations (C_{16}–C_{18} internal olefin) defined above in this table.

21. In § 435.14, footnote 2 to the table is revised to read as follows:

§ 435.14 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

* * * * *

BAT EFFLUENT LIMITATIONS

* * * *

[2] As determined by the static sheen test. See § 435.11(hh).

* * * * *

22. In § 435.15, footnotes 2, 3, and 5 through 11 to the table are revised to read as follows:

§ 435.15 Standards of performance for new sources (NSPS).

* * * * *

NEW SOURCE PERFORMANCE STANDARDS

* * * * *

[2] As determined by the suspended particulate phase (SPP) toxicity test. See § 435.11(gg).

[3] As determined by the static sheen test. See § 435.11(hh).

* * * * *

[5] PAH mass ratio = Mass (g) of PAH (as phenanthrene)/Mass (g) of stock base fluid as determined by EPA Method 1654, Revision A, [specified at § 435.11(u)] entitled "PAH Content of Oil by HPLC/UV," December 1992, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu).

[6] Base fluid sediment toxicity ratio = 10-day LC_{50} of C_{16}–C_{18} internal olefin/10-day LC_{50} of stock base fluid as determined by ASTM E 1367–92 [specified at § 435.11(ee)] method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, after preparing the sediment according to the procedure specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu).

[7] Biodegradation rate ratio = Cumulative headspace gas production (ml) of C_{16}–C_{18} internal olefin/Cumulative headspace gas production (ml) of stock base fluid, both at 275 days as determined by EPA Method 1647, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(e) and (uu).

[8] Drilling fluid sediment toxicity ratio = 4-day LC_{50} of C_{16}–C_{18} internal olefin drilling fluid/4-day LC_{50} of drilling fluid removed from drill cuttings at the solids control equipment as determined by ASTM E 1367–92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(ee) and (uu).

[9] As determined before drilling fluids are shipped offshore by the GC/MS compliance assurance method (EPA Method 1655), and as determined prior to discharge by the RPE method (EPA Method 1670) applied to drilling fluid removed from drill cuttings. If the operator wishes to confirm the results of the RPE method (EPA Method 1670), the operator may use the GC/MS compliance assurance method (EPA Method 1655). Results from the GC/MS compliance assurance method (EPA Method 1655) shall supersede the results of the RPE method (EPA Method 1670). EPA Method 1655 and 1670 are published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu).

[10] Maximum permissible retention of non-aqueous drilling fluid (NAF) base fluid on wet drill cuttings averaged over drilling intervals using NAFs as determined by EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu). This limitation is applicable for NAF base fluids that meet the base fluid sediment toxicity ratio (Footnote 6), biodegradation rate ratio (Footnote 7), PAH, mercury, and cadmium stock limitations (C_{16}–C_{18} internal olefin) defined above in this table.

[11] Maximum permissible retention of non-aqueous drilling fluid (NAF) base fluid on wet drill cuttings average over drilling intervals using NAFs as determined by EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(uu). This limitation is applicable for NAF base fluids that meet the ester base fluid sediment toxicity ratio and ester biodegradation rate ratio stock limitations defined as:

(a) Ester base fluid sediment toxicity ratio = 10-day LC_{50} of C_{12}–C_{14} ester or C_8 ester/10-day LC_{50} of stock base fluid as determined by ASTM E 1367–92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in EPA Method 1646, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(ee) and (uu);

(b) Ester biodegradation rate ratio = Cumulative headspace gas production (ml) of C_{12}–C_{14} ester or C_8 ester/Cumulative headspace gas production (ml) of stock base fluid, both at 275 days as determined by EPA Method 1647, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See § 435.11(e) and (uu); and (c) PAH mass ratio (Footnote 5), mercury, and cadmium stock limitations (C_{16}–C_{18} internal olefin) defined above in this table.

23. Subpart A of part 435 is amended by removing Appendices 1 through 7.

24. Subpart A of part 435 is amended by redesignating Appendix 8 as Appendix 1.

Subpart D—Coastal Subcategory

25. Section 435.41 is amended,

a. By revising paragraph (d).
b. By revising paragraph (e).
c. By revising paragraph (k).
d. By revising paragraph (m)(2).
e. By revising paragraph (q).
f. By revising paragraph (r).
g. By revising paragraph (y).
h. By revising paragraph (ee).
i. By revising paragraph (ff).
j. By adding paragraph (mm).

§ 435.41 Specialized definitions.

* * * * *

(d) *Base fluid retained on cuttings* as applied to BAT effluent limitations and NSPS refers to the "Determination of the Amount of Non-Aqueous Drilling Fluid (NAF) Base Fluid from Drill Cuttings by a Retort Chamber (Derived from API Recommended Practice 13B–2)", EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section.

(e) *Biodegradation rate* as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the "Protocol for the Determination of Degradation of Non Aqueous Base Fluids in a Marine Closed Bottle Biodegradation Test System: Modified ISO 11734:1995," EPA Method 1647, supplemented with "Procedure for Mixing Base Fluids With Sediments," EPA Method 1646. Both EPA Method 1646 and 1647 are published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section.

* * * * *

(k) *Diesel oil* refers to the grade of distillate fuel oil, as specified in the American Society for Testing and Materials Standard Specification for Diesel Fuel Oils D975–91, that is typically used as the continuous phase in conventional oil-based drilling fluids. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Copies may be inspected at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.* A copy may also be inspected at EPA's Water Docket, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

* * * * *

(m) * * *

(2) *Dry drill cuttings* means the residue remaining in the retort vessel after completing the retort procedure specified in EPA Method 1674, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section.

* * * * *

(q) *Formation oil* means the oil from a producing formation which is detected in the drilling fluid, as determined by the GC/MS compliance assurance method, EPA Method 1655, when the drilling fluid is analyzed before being shipped offshore, and as determined by the RPE method, EPA Method 1670, when the drilling fluid is analyzed at the offshore point of discharge. The GC/MS compliance assurance method and the RPE method approved for use with this part are published in the "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section. Detection of formation oil by the RPE method may be confirmed by the GC/MS compliance assurance method, and the results of the GC/MS compliance assurance method shall supersede those of the RPE method.

(r) *Garbage* means all kinds of victual, domestic, and operational waste, excluding fresh fish and parts thereof, generated during the normal operation of coastal oil and gas facility and liable to be disposed of continuously or periodically, except dishwater, graywater, and those substances that are defined or listed in other Annexes to MARPOL 73/78. A copy of MARPOL may be inspected at EPA's Water Docket, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

* * * * *

(y) *No discharge of free oil* means that waste streams may not be discharged that contain free oil as evidenced by the monitoring method specified for that particular stream, *e.g.,* deck drainage or miscellaneous discharges cannot be discharged when they would cause a film or sheen upon or discoloration of the surface of the receiving water; drilling fluids or cuttings may not be discharged when they fail EPA Method 1617 (Static Sheen Test), which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section.

* * * * *

(ee) *SPP toxicity* as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the bioassay test procedure, "Suspended Particulate Phase (SPP) Toxicity Test," presented in EPA Method 1619, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section.

(ff) *Static sheen test* means the standard test procedure that has been developed for this industrial subcategory for the purpose of demonstrating compliance with the requirement of no discharge of free oil. The methodology for performing the static sheen test is presented in EPA Method 1617, which is published in "Analytic Methods for the Oil and Gas Extraction Point Source Category," EPA–821–R–09–013. See paragraph (mm) of this section.

* * * * *

(mm) *Analytic Methods for the Oil and Gas Extraction Point Source Category* is the EPA document, EPA–821–R–09–013, that compiles analytic methods for this category. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be inspected at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.* A copy may also be inspected at EPA's Water Docket, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

26. In § 435.42, footnote 1 to the table is revised to read as follows:

§ 435.42 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

* * * * *

[1] No discharge of free oil. See § 435.41(y).

* * * * *

27. In § 435.43, footnotes 2 and 4 are revised to read as follows:

§ 435.43 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

* * * * *

BAT EFFLUENT LIMITATIONS

* * * * *

[2] As determined by the static sheen test. See § 435.41(ff).

* * * * *

[4] As determined by the suspended particulate phase (SPP) toxicity test. See § 435.41(ee).

* * * * *

28. In § 435.44 footnote 2 to the table is revised to read as follows:

§ 435.44 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

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BAT EFFLUENT LIMITATIONS

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[2] As determined by the static sheen test. See § 435.41(ff).

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29. In § 435.45, footnotes 2 and 4 to the table are revised to read as follows:

§ 435.45 Standards of performance for new sources (NSPS).

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NSPS EFFLUENT LIMITATIONS

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[2] As determined by the static sheen test. See § 435.41(ff).

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[4] As determined by the suspended particulate phase (SPP) toxicity test. See § 435.41(ee).

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