NATIONAL ARCHIVES AND RECORDS ADMINISTRATION
1985

Federal Register

Tuesday,
April 6, 2004

Part II

Environmental Protection Agency

40 CFR Part 122, et al.

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Proposed Rule

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 122, 136, 141, 143, 403, 430, 455, and 465

[FRL–7638–9]

RIN 2040–AD71

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule.

SUMMARY: EPA is proposing changes to analysis and sampling procedures in wastewater regulations. These changes include proposal of vendor-developed methods; new EPA and voluntary consensus standard bodies (VCSB) methods; updated versions of currently approved methods; revised method modification and analytical requirements; withdrawal of certain outdated methods; and changes to sample collection, preservation, and holding time requirements. EPA also is proposing changes to drinking water analysis and monitoring. These changes include proposal of vendor-developed methods; new EPA and VCSB methods; and updated VCSB methods. The addition of new and updated methods to the wastewater and drinking water regulations will provide increased flexibility to the regulated community and laboratories in the selection of analytical methods. Finally, EPA is soliciting comment on the guidance document *EPA Microbiological Alternate Test Procedure (ATP) Protocol for Drinking Water, Ambient Water, and Wastewater Monitoring Methods.*

DATES: Comments must be postmarked, delivered by hand, or electronically mailed on or before June 7, 2004. Comments provided electronically will be considered timely if they are submitted electronically by 11:59 p.m. Eastern Time on June 7, 2004.

ADDRESSES: Comments may be submitted by mail to Water Docket, U.S. Environmental Protection Agency (4101T), 1200 Pennsylvania Avenue NW., Washington DC 20460, or electronically through EPA Dockets at *http://www.epa.gov/edocket/,* Attention Docket ID No. OW–2003–0070. See Section C of the **SUPPLEMENTARY INFORMATION** section for additional ways to submit comments and more detailed instructions.

FOR FURTHER INFORMATION CONTACT: For information regarding the proposed changes to wastewater regulations contact Marion Kelly, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave., NW., Washington, DC 20460, 202–566–1045 (e-mail: *Kelly.Marion@epa.gov*). For information regarding the proposed changes to drinking water regulations, contact Herbert J. Brass, Technical Support Center (MS 140), USEPA, Office of Ground Water and Drinking Water, 26 West Martin Luther King Drive, Cincinnati, OH 45268, 513–569–7936 (e-mail: *Brass.Herb@epa.gov*). For information on the guidance document *EPA Microbiological Alternate Test Procedure (ATP) Protocol for Drinking Water, Ambient Water, and Wastewater Monitoring Methods* contact Robin K. Oshiro, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave., NW., Washington, DC 20460, 202–566–1075 (e-mail: *Oshiro.Robin@epa.gov*).

SUPPLEMENTARY INFORMATION:

A. Potentially Regulated Entities

1. Clean Water Act

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits that comply with the technology-based and water quality-based requirements of the Clean Water Act (CWA). In doing so, NPDES permitting authorities, including States, Territories, and Tribes, make several discretionary choices when they write a permit. These choices include the selection of pollutants to be measured and, in many cases, limited in permits. If EPA has "approved'' (i.e., promulgated through rulemaking) procedures for analysis of pollutants (i.e., test procedures), the NPDES permit must include one of the approved testing procedures or an approved alternate test procedure. Similarly, if EPA has approved sampling requirements, measurements taken under an NPDES permit must comply with these requirements. Therefore, entities with NPDES permits could potentially be regulated by the proposed actions in this rulemaking. In addition, when an authorized State, Territory, or Tribe certifies Federal licenses under CWA section 401, they must use the standardized analysis and sampling procedures. Categories and entities that could potentially be regulated 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 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 122.1, (NPDES purpose and scope), 40 CFR 136.1 (NPDES permits and CWA), 40 CFR 403.1 (Pretreatment standards purpose and applicability), 40 CFR 430.00 (Pulp, paper, and paperboard point source category applicability), 40 CFR 455.20, 455.30, 455.40, 455.60 (Pesticide point source category applicability), and 40 CFR 465.01 (Coil coating point source category 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.

2. Safe Drinking Water Act

Public water systems are the regulated entities required to conduct analyses to measure for contaminants in water samples. However, EPA Regions, as well as States, and Tribal governments with primacy to administer the regulatory program for public water systems under the Safe Drinking Water Act, sometimes conduct analyses to measure for contaminants in water samples. If EPA has established a maximum contaminant level ("MCL'') for a given drinking water contaminant, the Agency also approves (*i.e.*, promulgates through rulemaking) standardized testing procedures for analysis of the contaminant. Once EPA standardizes such test procedures, analysis using a standard test procedure (or approved alternate test procedures) is generally required. Public water systems required to test water samples must use one of the approved standardized test procedures. Categories and entities that may ultimately be regulated include:

Category	Examples of potentially regulated entities	NAICS1[1]
State, Local, & Tribal Governments	States, local and tribal governments that analyze water samples on behalf of public water systems required to conduct such analysis; States, local and tribal governments that themselves operate community and non-transient non-community water systems required to monitor.	924110
Industry	Private operators of community and non-transient non-community water systems required to monitor.	221310
Municipalities	Municipal operators of community and non-transient non-community water systems required to monitor.	924110

[1] North American Industry Classification System.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be regulated by this action. The table lists types of entities that EPA is now aware could potentially be regulated by this action. Other types of entities not listed in the tables could also be regulated. To determine whether your facility is regulated by this action, you should carefully examine the applicability language at 40 CFR 141.2 (definition of public water system). 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. How Can I Get Copies of This Document and Other Related Information?

1. Docket

EPA has established an official public docket for this action under Docket ID No. OW–2003–0070. The official public docket consists of the documents specifically referenced in this action, any public comments received, and other information related to this action. Although a part of the official docket, the public docket does not include Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. The official public docket is the collection of materials that is available for public viewing at the Water Docket in the EPA Docket Center, EPA West Building, Room B102, 1301 Constitution Avenue NW., Washington, DC. The EPA Docket Center 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 access to docket materials, please call ahead to schedule an appointment. Every user is entitled to copy 266 pages per day before incurring a charge. The Docket may charge 15 cents per page for each page over the page limit plus an administrative fee of $25.00.

2. Electronic Access

You may access this **Federal Register** document electronically through the EPA Internet under the "**Federal Register**" listings at *http://www.epa.gov/fedrgstr/.* An electronic version of the public docket is available through EPA's electronic public docket and comment system, EPA Dockets. You may use EPA Dockets at *http://www.epa.gov/edocket/* to submit or view public comments, access the index listing of the contents of the official public docket, and to access those documents in the public docket that are available electronically. Once in the system, select "search,'' then key in the appropriate docket identification number.

Certain types of information will not be placed in the EPA Dockets. Information claimed as CBI and other information whose disclosure is restricted by statute, which is not included in the official public docket, will not be available for public viewing in EPA's electronic public docket. EPA's policy is that copyrighted material will not be placed in EPA's electronic public docket but will be available only in printed, paper form in the official public docket. Although not all docket materials may be available electronically, you may still access any of the publicly available docket materials through the docket facility identified in Section B.1.

For public commenters, it is important to note that EPA's policy is that public comments, whether submitted electronically or in paper, will be made available for public viewing in EPA's electronic public docket as EPA receives them and without change, unless the comment contains copyrighted material, CBI, or other information for which disclosure is restricted by statute. When EPA identifies a comment containing copyrighted material, EPA will provide a reference to that material in the version of the comment that is placed in EPA's electronic public docket. The entire printed comment, including the copyrighted material, will be available in the public docket.

Public comments submitted on computer disks that are mailed or delivered to the docket will be transferred to EPA's electronic public docket. Public comments that are mailed or delivered to the Docket will be scanned and placed in EPA's electronic public docket. Where practical, physical objects will be photographed, and the photograph will be placed in EPA's electronic public docket along with a brief description written by the docket staff.

C. How and To Whom Do I Submit Comments?

You may submit comments electronically, by mail, or through hand delivery/courier. To ensure proper receipt by EPA, identify the appropriate docket identification number in the subject line on the first page of your comment. Please ensure that your comments are submitted within the specified comment period. Comments received after the close of the comment period will be marked "late.'' EPA is not required to consider these late comments.

1. Electronically

If you submit an electronic comment as prescribed below, EPA recommends that you include your name, mailing address, and an e-mail address or other contact information in the body of your comment. Also include this contact information on the outside of any disk or CD ROM you submit, and in any cover letter accompanying the disk or CD ROM. This ensures that you can be identified as the submitter of the comment and allows EPA to contact you in case EPA cannot read your comment due to technical difficulties or needs further information on the substance of your comment. EPA's policy is that EPA will not edit your comment, and any identifying or contact information provided in the body of a comment will be included as part of the comment that is placed in the official public docket, and made available in EPA's electronic public docket. 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.

i. *EPA Dockets.* Your use of EPA's electronic public docket to submit comments to EPA electronically is EPA's preferred method for receiving comments. Go directly to EPA Dockets at *http://www.epa.gov/edocket,* and follow the online instructions for submitting comments. Once in the system, select "search,'' and then key in Docket ID No. OW–2003–0070. The system is an "anonymous access'' system, which means EPA will not know your identity, e-mail address, or other contact information unless you provide it in the body of your comment.

ii. *E-mail.* Comments may be sent by electronic mail (e-mail) to: *OW-docket@epamail.epa.gov,* Attention Docket ID No. OW–2003–0070. In contrast to EPA's electronic public docket, EPA's e-mail system is not an "anonymous access'' system. If you send an e-mail comment directly to the Docket without going through EPA's electronic public docket, EPA's e-mail system automatically captures your e-mail address. E-mail addresses that are automatically captured by EPA's e-mail system are included as part of the comment that is placed in the official public docket, and made available in EPA's electronic public docket.

iii. *Disk or CD ROM.* You may submit comments on a disk or CD ROM that you mail to the mailing address identified in Section C.2. These electronic submissions will be accepted in WordPerfect or ASCII file format. Avoid the use of special characters and any form of encryption.

2. By Mail

Send an original and three copies of your comments to Water Docket, U.S. Environmental Protection Agency (4101T), 1200 Pennsylvania Avenue NW., Washington, DC 20460, Attention Docket ID No. OW–2003–0070.

3. By Hand Delivery or Courier

Deliver your comments to the Water Docket in the EPA Water Center, EPA West Building, Room B102, 1301 Constitution Avenue NW., Washington, DC, Attention Docket ID No. OW–2003–0070. Such deliveries are only accepted during the Docket's normal hours of operation as identified in Section B.1.

D. How Should I Submit CBI to the Agency?

Do not submit information that you consider to be CBI electronically through EPA's electronic public docket or by e-mail. You may claim information that you submit to EPA as CBI by marking any part or all of that information as CBI (if you submit CBI on disk or CD ROM, 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 CBI). Information so marked will not be disclosed except in accordance with procedures set forth in 40 CFR part 2.

In addition to one complete version of the comment that includes any 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 and EPA's electronic public docket. If you submit the copy that does not contain CBI on disk or CD ROM, mark the outside of the disk or CD ROM clearly that it does not contain CBI. Information not marked as CBI will be included in the public docket and EPA's electronic public docket without prior notice. If you have any questions about CBI or the procedures for claiming CBI, please consult the person identified in the **FOR FURTHER INFORMATION CONTACT** section.

E. What Should I Consider as I Prepare My Comments for EPA?

You may find the following suggestions helpful for preparing your comments:

1. Explain your views as clearly as possible.
2. Describe any assumptions that you used.
3. Provide any technical information and/or data you used that support your views.
4. If you estimate potential burden or costs, explain how you arrived at your estimate.
5. Provide specific examples to illustrate your concerns.
6. Offer alternatives.
7. Make sure to submit your comments by the comment period deadline.
8. To ensure proper receipt by EPA, identify the appropriate docket identification number in the subject line on the first page of your response. It would also be helpful if you provided the name, date, and **Federal Register** citation related to your comments.

F. Abbreviations and Acronyms Used in the Preamble and Proposed Rule

ASTM: ASTM International
ATP: Alternate Test Procedure
CIE–UV: Capillary Ion Electrophoresis with Indirect Ultraviolet Detection
CWA: Clean Water Act
EMMC: Environmental Monitoring Management Council
EPA: Environmental Protection Agency
FLAA: Flame Atomic Absorption Spectroscopy
GC–MS: Gas Chromatography with Mass Spectrometry Detection
IC: Ion Chromatography
ICP–AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP–MS: Inductively Coupled Plasma-Mass Spectroscopy
ISE: Ion Selective Electrode
NPDES: National Pollutant Discharge Elimination System
NPDWR: National Primary Drinking Water Regulations
NSDWR: National Secondary Drinking Water Regulations
SDWA: Safe Drinking Water Act
STGFAA: Stabilized Temperature Graphite Furnace Atomic Absorption Spectroscopy

Table of Contents

G. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks
H. Executive Order 13211: Actions that Significantly Affect Energy Supply, Distribution, or Use
I. National Technology Transfer and Advancement Act

I. Statutory Authority

A. Clean Water Act

EPA is proposing this action 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 publishes test procedure regulations (including analysis and sampling requirements) for CWA programs at 40 CFR part 136, though some specific requirements are in other sections (*e.g.*, 40 CFR Chapter I, Subchapters N and O).

B. Safe Drinking Water Act

The Safe Drinking Water Act (SDWA), as amended in 1996, requires EPA to promulgate national primary drinking water regulations (NPDWRs) that specify maximum contaminant levels (MCLs) or treatment techniques for drinking water contaminants (SDWA section 1412 (42 U.S.C. 300g–1)). NPDWRs apply to public water systems pursuant to SDWA sections 1401(1)(A) (42 U.S.C. 300f(1)(A)). According to SDWA section 1401(1)(D), NPDWRs include ''* * * criteria and procedures to assure a supply of drinking water which dependably complies with such maximum contaminant levels; including accepted methods for quality control and testing procedures * * *'' (42 U.S.C. 300f(1)(D)). In addition, SDWA section 1445(a) authorizes the Administrator to establish regulations for monitoring to help determine whether persons are acting in compliance with the requirements of the SDWA (42 U.S.C. 300j–4). EPA's promulgation of analytical methods for NPDWRs is authorized under these sections of the SDWA as well as the general rulemaking authority in SDWA section 1450(a) (42 U.S.C. 300j–9(a)).

The SDWA also authorizes EPA to promulgate national secondary drinking water regulations (NSDWRs) for contaminants in drinking water that primarily affect the aesthetic qualities relating to the public acceptance of drinking water (SDWA section 1412 (42 U.S.C. 300g–1)). These regulations are not Federally enforceable but are guidelines for the States (40 CFR 143.1). The NSDWRs also include analytical techniques for determining compliance with the regulations (40 CFR 143.4). EPA's promulgation of analytical methods for NSDWRs is authorized under general rulemaking authority in SDWA section 1450(a) (42 U.S.C. 300j–9(a)).

II. Explanation of Today's Action

A. Methods for NPDES Compliance Monitoring

EPA approves analytical methods for measuring regulated pollutants in wastewater. Regulated and regulatory entities use these approved methods for determining compliance with an NPDES permit or other monitoring requirement. Often, these entities have a choice in deciding which approved method they will use to measure a pollutant because multiple approved methods are available.

This rule proposes to add to the list of approved test procedures for a number of pollutants. Some proposed methods introduce new technologies to the NPDES program, while others are updated versions of previously approved methods. EPA believes that these additions will improve data quality and provide the regulated community with greater flexibility. Furthermore, many of the additions will promote consistency between the NPDES (wastewater) and NPDWRs/NSDWRs (drinking water) compliance monitoring programs by adopting the same versions of methods for both programs—allowing laboratories to use a single version of a method to satisfy multiple water compliance monitoring needs.

This rule also proposes to allow increased method flexibility by explicitly allowing certain technical changes (*e.g.*, allowing the use of capillary columns in gas chromatography methods, allowing the addition of salts—under certain conditions—to resolve interferences in extraction procedures). Finally, this rule proposes to remove certain outdated methods, including chlorofluorocarbon-based oil and grease methods.

B. Sampling, Sample Preservation, and Holding Times for NPDES Compliance Monitoring

EPA publishes sampling, sample preservation, and holding time requirements in regulations to help ensure that samples collected for NPDES compliance monitoring provide a representative measurement of the pollutants in wastestreams. This rule proposes to update these requirements to reflect new information and create consistency among CFR sections.

C. Editorial Changes to 40 CFR Part 136

This rule proposes editorial changes to 40 CFR part 136 to correct errors and update information.

D. Methods for NPDWR and NSDWR Compliance Monitoring and Monitoring Requirements

EPA approves analytical methods for monitoring contaminants in drinking water. The drinking water industry uses these approved methods for determining compliance with NPDWRs and NSDWRs. Because multiple methods are generally available, public water systems often have a choice in deciding which approved method they will use to measure a drinking water contaminant.

This rule proposes to add a new means of monitoring for compliance with a radiological contaminant limit, and new methods for chemical contaminant monitoring. These additions will provide greater monitoring flexibility.

E. Microbiological ATP Protocol

EPA is soliciting comments on ''EPA Microbiological Alternate Test Procedure (ATP) Protocol for Drinking Water, Ambient Water, and Wastewater Monitoring Methods—Guidance'' (July 2003; EPA–821–B–03–004) (Protocol).

III. Summary of Proposed Revisions to Wastewater Regulations

A. Analytical Methods for NPDES Compliance Monitoring

1. Chemical Alternate Test Procedures

To promote method flexibility, EPA maintains a program whereby stakeholders (*e.g.*, instrument manufacturers, environmental laboratories, regulated entities) can apply for EPA approval of alternate test procedures. The Alternate Test Procedure (ATP) program is codified at 40 CFR 136.4 and 136.5 for wastewater. This rule proposes to approve three alternate test procedures at 40 CFR part 136 for monitoring chemical pollutants:

- ''Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion

Electrophoresis and Chromate Electrolyte'' (D6508, Rev. 2) by Waters Corporation.

- "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and determination of cyanide by flow injection analysis'' (QuikChem Method 10–204–00–1–X) by Lachat Instruments.
- "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate'' (Kelada-01) by Dr. Nabih Kelada.

This rule also proposes to approve one method in the pulp, paper, and paperboard point source category regulations at 40 CFR 430.02:

- "Chlorinated Phenolics in Water by *In situ* Acetylation and GC/MS Determination'' (Method CP–86.07) by the National Council for Air and Stream Improvement (NCASI).

Each of the above-listed ATPs offers substantial advantages over currently approved methods, and their approval will give analysts additional flexibility in meeting monitoring requirements.

a. Anions by Capillary Ion Electrophoresis With Indirect Ultraviolet Detection (CIE–UV)

Waters Corporation's "Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte'' (Method D6508, Rev. 2) is a new method that uses capillary ion electrophoresis to determine common anions—bromide, chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate—in drinking water and wastewater. Method D6508 appears to provide an acceptable technological alternative to ion chromatography and wet chemical methods in terms of method performance and is equivalent to other approved methods in the working range. In addition, the method is relatively easy to use (the CIE–UV system has fewer moving parts and components than an ion chromatography system making it easier to operate and maintain), involves relatively low cost equipment (the cost of a CIE–UV capillary column is $30 compared to an ion chromatography column that can be greater than $800), and generally reduces laboratory wastes (less than 100 milliliters of waste is generated daily).

Capillary ion electrophoresis (CIE) employs the same general principle of "separation followed by detection'' common to all chromatography methods. Anions migrate through a silica capillary column containing an electrolyte solution under the influence of an electric field. With CIE, anions in the sample separate according to their equivalent ionic conductance and mobility, and are measured using indirect ultraviolet (UV) detection: the UV absorbing electrolyte anion is displaced charge-for-charge by analyte anion. The corresponding decrease in background absorption is proportional to the concentrations of anions.

An 11-laboratory validation study characterized the performance of Method D6508. Eight concentration levels included analyses of reagent water, "substitute'' wastewater, "real'' wastewater, and drinking water matrix types. The range of Method D6508 is 0.2 to 50 mg/L for all analytes but fluoride, for which the range is 0.2 to 25 mg/L. The method provides precision and recovery data for all analytes in all matrices. For example, across all analytes at concentrations of approximately 3 mg/L in real wastewater, the range of multilaboratory recovery was 84–100%, and relative standard deviation was 6–26%. Waters Corp. generated quality control limits in the method from the study data available in the docket supporting this proposal.

ASTM International (ASTM) adopted a previous version of Method D6508. If the updated version of the method included in the docket is adopted by ASTM prior to publication of the final rule, the ASTM method also may be approved.

A copy of Method D6508 and the method validation study report are in the docket for this proposed rule. In addition, copies of Method D6508 may be obtained from Waters Corporation. Contact: Jim Krol, Waters Corp., 34 Maple St, Milford, MA 01757, 508/482–2131 (Office), 508/482–3625 (FAX), and *Jim_Krol@Waters.com.*

b. Cyanide Microdistillation

Lachat Instrument's "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis'' (QuikChem Method 10–204–00–1–X) is a method that determines total cyanide in drinking water and wastewater. The method employs the MICRO DIST distillation apparatus, a reduced-volume and disposable counterpart to other distillation apparatuses. MICRO DIST substantially reduces distillation time (by 50% as compared with the currently approved macrodistillation) and laboratory wastes (because it requires small sample and reagent volumes required). It easily allows multiple simultaneous distillations (one distillation heating block accommodates 21 MICRO DIST apparatuses). MICRO DIST also has lower costs than some other cyanide distillations due to time saved (in analysis and sample throughput) and reduced waste disposal costs.

Using MICRO DIST, total cyanide is determined by distilling the sample and measuring cyanide generated using a technique for cyanide ion detection (*e.g.*, colorimetry). Six milliliters of sample are added to a distillation tube along with standard cyanide distillation reagents (sulfuric acid, magnesium chloride). A cyanide collector tube, which consists of a gas permeable membrane and sodium hydroxide absorber solution, is attached to the distillation tube; the distillation and collector tubes together comprise the MICRO DIST apparatus. The sample is heated for one-half hour, during which hydrogen cyanide gas distills from the sample, passes through the gas permeable membrane, and collects in the sodium hydroxide absorber solution. QuikChem Method 10–204–00–1–X provides instructions for measuring cyanide in the absorber solution using an automated colorimeter. However, the Method specifies that the absorber solution may be analyzed using another procedure (*e.g.*, manual colorimetry) as well, provided all requirements in the Method are followed (*e.g.*, pH of the absorber solution and standards are adjusted to match). This rule proposes both Method 10–204–00–1–X as a stand alone method, and the MICRO DIST distillation procedure found in that method as an alternative to other approved distillation procedures.

Method performance was characterized by two single laboratory studies, and a nine-laboratory validation study. Lachat and the Research Triangle Institute performed single laboratory studies that demonstrated that recovery of complex cyanides using MICRO DIST was equivalent to recovery with a conventional distillation apparatus. The nine-laboratory validation study demonstrated that Method 10–204–00–1–X with the MICRO DIST apparatus provided equivalent performance to EPA-approved total cyanide methods across laboratories.

In validation of MICRO DIST, EPA reviewed data on recoveries of free cyanide from wastewater matrices, and the recovery of complex cyanides. EPA did not receive data on the recovery of particulate cyanide, but other factors suggest that particulate cyanide will not pose a problem with the method. These factors include (1) the performance of the method in recovering complex cyanides, (2) the increasing reagent concentrations in the solution during distillation (due to sample transfer during distillation), and (3) the fact that

MICRO DIST employs a hard distillation versus a reflux distillation. EPA requests comments on the issue of the distillation and solicits data regarding MICRO DIST's recovery of particulate cyanide.

A copy of QuikChem Method 10–204–00–1–X and the method validation study report are in the docket for this proposed rule. In addition, copies of QuikChem Method 10–204–00–1–X may be obtained from Lachat Instruments, 6645 W. Mill Rd., Milwaukee, WI 53218, USA. Phone: 414–358–4200.

c. Cyanide by UV-digestion/Flash Distillation/Colorimetry

Dr. Nabih Kelada's "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate,'' EPA 821–B–01–009 (Kelada-01) is an automated procedure that determines total cyanide and acid dissociable cyanide in drinking water and wastewater. The procedure uses a two-stage sample digestion system to determine total cyanide. A sample is introduced into a flow analysis system. The sample then passes through an irradiation coil, where it is exposed to intense ultraviolet (UV) light from a high power (*e.g.,* 550 Watt) UV photochemical bulb. The UV light breaks down cyanide complexes (including strong ferro- and ferri-cyanide complexes) to free cyanide. The irradiated sample containing free cyanide then passes though a distillation coil from which the free cyanide is distilled into a flow colorimetry system (similar to that used in EPA Methods 335.3 and 335.4), and cyanide concentration is determined. All complex cyanides recovered using the total cyanide manual distillation are recovered using Kelada-01.

When the irradiation coil is by-passed—exposing sample only to a distillation coil—"acid dissociable'' cyanide is determined. The complexes measured are equivalent to those measured using cyanide amenable to chlorination (CATC) and "available'' cyanide procedures, according to single laboratory studies performed by the Metropolitan Water Reclamation District of Greater Chicago.

Kelada-01 offers a number of substantial advantages over currently approved methods, such as a reduced analysis time (from one to two hours to minutes), and substantially reduced effects of many interferences encountered with manual distillation methods. Kelada-01 also produced very precise and accurate results, as demonstrated in single laboratory validation studies by the Metropolitan Water Reclamation District of Greater Chicago, an interlaboratory study managed by Environment Canada, and an ASTM "round-robin'' (interlaboratory) validation study. These studies generally showed total and acid dissociable cyanide recoveries from samples between 90% and 110%, and relative standard deviations of less than 10%. The reported lower limit of detection is 0.5 µg/L.

For determination of total cyanide, Kelada-01 can be configured to use UV-irradiation under alkaline conditions (alkaline mode) or acidic conditions (acidic mode). EPA has reviewed data on recoveries of free cyanide and complex cyanides from a variety of wastewater matrices in both modes. Given the successful recovery of cyanide complexes from a variety of effluents, opacity does not appear to effect the recovery of cyanide complexes. In addition, side-by-side comparative data on high particulate samples (*e.g.,* sludge) in the article *Automated Direct Measurements of Total Cyanide Species and Thiocyanate, and their Distribution in Wastewater and Sludge* (Journal WPCF, 61–3, pp. 350–56, March 1989) demonstrating Kelada-01's superior recovery of cyanide (relative to manual distillation) when running in alkaline mode, supporting the conclusion that particulate cyanide recovery is not a concern with this method. Corresponding data for use in acidic mode is not available. However, EPA requests comment on whether the use of Kelada-01 for determination of total cyanide should be restricted to alkaline mode or should allow determinations in both alkaline and acidic mode.

A copy of Kelada-01 and the method validation study report are in the docket for this proposed rule. In addition, copies of Kelada-01 are available from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. Phone: 800–553–6847.

d. Phenolics in Bleach Plant Filtrate by Gas Chromatography–Mass Spectrometry (GC–MS)

NCASI Method CP–86.07, "Chlorinated Phenolics in Water by *In situ* Acetylation and GC/MS Determination'' (January 2002) for determining chlorinated phenols, chlorinated guaiacols, chlorinated catechols, chlorinated benzaldehydes (*i.e.,* vanillins and syringaldehydes), and trichlorsyringol in bleach plant filtrate is an ATP to EPA Method 1653. The complete list of analytes to which Method CP–86.07 is applicable is provided in the method.

A 300-mL aliquot of aqueous sample is spiked with internal standards and surrogates and is treated to form phenolate ions at a pH of 9 to 11.5. The phenolate ions are converted *in situ* (*i.e.,* in the aqueous matrix) to their acetate derivatives which are then extracted with hexane. The extract is analyzed using GC/MS.

EPA is proposing Method CP–86.07 specifically for use under the regulations at 40 CFR part 430 (for the pulp and paper industry). NCASI Method CP–86.07 was developed to reduce analytical costs (by $200 to $300 per analysis) and the need for several isotopically labeled standards, reduce sample and reagent volumes (*e.g.,* sample volume is reduced over 300% from EPA Method 1653), and addresses certain interferences in pulp and paper effluent. With regard to performance, participants in a four-laboratory validation study met the quality control (QC) acceptance criteria specified in EPA Method 1653, demonstrating equivalent performance.

NCASI Method CP–86.07 is available from the Publications Coordinator, NCASI, P.O. Box 13318, Research Triangle Park, NC 27709–3318. Phone 919–588–1987. You can also find a copy of the method and the validation study report in the docket for this proposed rule.

2. Whole Effluent Toxicity Alternate Test Procedure—Microtox® 1010

By today's notice, EPA invites comments on whether to approve a standardized testing procedure measuring acute toxicity of aqueous test samples to bacteria. Specifically, in response to a request from Strategic Diagnostics, Inc. (SDI), EPA requests comment on whether to approve, under 40 CFR part 136, SDI's "Method for Measuring the Acute Toxicity of Wastewater and Receiving Water with the *Vibrio fischeri* (NRRL B–11177) Microtox® Test System'' (Microtox® 1010) for determining the acute toxicity of wastewater, receiving waters, and other aqueous samples. In this test, multiple, one-milliliter sample dilutions (minimum of five) are added to sample wells containing about one million saltwater bioluminescent bacteria, *Vibrio fischeri* strain NRRL B–11177. The bacterial bioluminescence is a by-product of cellular respiration correlating to organism health, and is measured in each well using a photometer. Measurements at the various dilutions, referenced against a control well, are taken over 15 minutes and used to calculate the concentration at which the organisms manifest a 50% effect (EC_{50}), in this case, a decrease in bioluminescence. The EC_{50} is the concentration of sample at which a 50% reduction in bioluminescence occurs, and it is analogous to the 50% inhibition concentration (IC_{50}) used in other toxicity tests.

Approval of Microtox® 1010 will add a new phylogenic category which can be used to measure toxicity, specifically bacteria. EPA proposes to approve the use of Microtox® 1010 to screen discharges to freshwater for acute toxicity. EPA invites comment on the appropriateness of including a bacteriological test to measure toxicity for regulation in permits. EPA also invites comments on two options for the use of Microtox® 1010 in discharges to marine and estuarine waters.

Advantages of Microtox® 1010 Relative to Other Toxicity Test Methods

Microtox® 1010 may be useful as a screening level test for discharges to both freshwater and marine and estuarine waters when it is used in conjunction with EPA's current whole effluent toxicity (WET) test methods.

Microtox® 1010 also may be useful as a definitive test for discharges to marine and estuarine waters. The Microtox® 1010 method employs a very large number of organisms (one million bacteria), and as such, the test is not influenced by the responses of a small number of test organisms. The method requires small sample volumes, making the collection and shipment of samples simpler and more affordable. The analysis time is short (15 minutes), and the method is relatively inexpensive ($50–$150, compared to $300–$600 for the currently approved acute toxicity tests).

While Microtox® 1010 would be an addition to the suite of EPA WET methods, the technology is not new. Extensive research and validation have been conducted using *Vibrio fischeri,* culminating in more than 350 peer-reviewed papers (including 17 authored by EPA staff) and adoption of ASTM Standard D5660–96, "Standard Test Method for Assessing the Microbial Detoxification of Chemically Contaminated Water and Soil Using a Toxicity Test with a Luminescent Bacterium.'' In response to previous EPA concerns about a lack of information on Microtox® 1010 (see a Supplementary Information Document [Response-to-Comments Document] from the 1995 WET rule [60 FR 53529, 53536; October 16, 1995]), SDI's predecessor, Azur Environmental, conducted a validation study (ATP Application SL97–0002).

Approval of Microtox® 1010 for measurement as a pollutant parameter under 40 CFR part 136 would allow toxicity evaluation to be expanded to an important phylogenetic group and trophic level that is not now addressed in the WET program. Bacteria are ecologically relevant links in nutrient and energy cycling and, consequently, are generally important to assessing the health of the environment.

EPA anticipates, however, there are some limitations for using the Microtox® 1010 method for WET testing which are discussed below.

Limitations

Adding Microtox® 1010 to the suite of WET test procedures poses challenges for the National Pollutant Discharge Elimination System (NPDES) WET program. First, Microtox® 1010 employs a saltwater bacterium, and therefore, the salinity of the samples to which the method is applied needs to be adjusted to near that of seawater to avoid artificial stressors to the test organisms. As such, the modified sample may not represent the characteristics of the actual effluent.

Second, Microtox® 1010 is less sensitive to some common contaminants (certain metals and ammonia) than the currently approved WET procedures. Conversely, Microtox® 1010 may be more sensitive than the currently approved WET procedures to other sources of toxicity, for example, sulfur and sulfur compounds. Currently, EPA guidance in the "Technical Support Document for Water Quality-based Toxics Control'' (TSD, 1991) recommends that initial effluent testing using species from three different phyla for the purpose of identifying the most sensitive test species. Bacteria would be a fourth phylum. EPA is concerned that economic pressure to implement a cheaper and quicker test (*e.g.*, Microtox® 1010) may cause some regulatory authorities to abandon the recommended guidance for initial testing and selection of the species that is most sensitive to the toxicity of a particular effluent. For these reasons, EPA also invites comment on the following uses of the Microtox® 1010 test to measure samples and protect water quality.

Use in Discharges to Freshwater

Using the Microtox® 1010 to evaluate discharges to freshwaters may not be appropriate, because the required salinity adjustment itself could affect the toxicity of the sample, and the salinity of the adjusted sample would not represent either the effluent being discharged or the receiving water.

Therefore, EPA invites comment on the use of Microtox® 1010 as a "screening'' test for freshwater. Under this approach, NPDES permits for discharges to freshwater would not set limits based on acute toxicity to bacteria determined by the Microtox® 1010 test. Instead, the test would be used to provide a "snapshot'' for toxicity potential of uncharacterized test samples to decide whether further toxicity evaluation seems warranted. For example, EPA anticipates the test would be very useful in situations where test samples display intermittent toxicity or for a toxicity reduction evaluation (TRE). Alternatively, operators of POTW pretreatment programs could use the test for rapid analysis of the toxicity of samples from users of the POTW. For these uses, EPA would not need to approve the Microtox® 1010 test for use in 40 CFR part 136.

Use in Discharges to Marine and Estuarine Water

EPA is considering two options for the use of Microtox® 1010.

Option (1)—Use as a Screening Procedure

Under this option, Microtox® 1010 would be implemented for discharges to marine and estuarine water in the same manner as proposed for discharges to freshwater. As such, EPA would not need to approve the test for use under 40 CFR part 136.

Option (2)—Use as a Definitive Test

Under this option, the Microtox® 1010 test would be used to establish NPDES permit limits (in lieu of other WET test procedures) if the Microtox® 1010 test organisms are the most sensitive in detecting toxicity of a given test sample. This option would rely on bacteria as an additional phylogenetic group by which to evaluate a sample's toxicity. To assist EPA in determining whether to incorporate bacteria as an additional phylogenetic group for which toxicity should be evaluated, the Agency invites comment on whether adjusting the salinity of discharges to marine and estuarine waters inappropriately introduces a variable to the measurement of acute toxicity.

EPA solicits comments on all aspects of the Microtox® 1010 proposal, but is particularly interested in comment on the following issues.

- What is the most appropriate for the use of Microtox® 1010 with marine and estuarine waters? A screening test? A definitive test? Both? Neither?
- Should the use of Microtox® 1010 be precluded where toxicity in discharges is known to be due primarily to metals and/or ammonia?

- Should EPA approve this proprietary test procedure? EPA does not generally endorse particular products or services. If EPA does approve use of Microtox® 1010 under 40 CFR part 136, how should the Agency reflect the essential attributes of the test that are not proprietary if it promulgates a final regulation approving the procedure?
- Will the regulated community require additional guidance from EPA regarding the implementation of Microtox® 1010 in the WET monitoring scheme?
- Should testing with Microtox® 1010 and three other species in the currently approved WET test procedures (*e.g.*, fish, invertebrates, and plants) be conducted quarterly for one year to address concerns of sensitivity to metals, ammonia, and/or unidentified toxicants?
- Are there additional bacteria-based methods that EPA should consider?

A copy of Microtox® 1010 and the method validation study report are in the docket for this proposed rule. In addition copies, of Microtox® 1010 are available from Strategic Diagnostics, Inc., 111 Pencander Drive, Newark, DE 19702–3322, Phone: 800–544–8881, Fax: 302–456–6789.

3. New Methods and Method Practices Proposed on October 18, 1995

The methods that EPA proposes to approve in NPDES regulations at 40 CFR part 136 include technologies that have been in use for many years. Many of the methods have been used in the NPDES monitoring program on a limited basis or have been used in other EPA programs (such as the NPDWR program). Among the methods EPA proposes to approve are a group of methods (or earlier revisions of the methods) that EPA initially proposed for approval on October 18, 1995 (60 FR 53987, hereinafter referred to as the "10–18 proposal''). Although EPA did not take final action on the 10–18 proposal, NPDES-approval for these methods has been granted to individual applicants on a case-by-case basis under the ATP program provisions at 40 CFR 136.4 and 136.5.

EPA proposes these methods again (along with updated equivalent methods), instead of publishing a final rule, because over eight years have passed since EPA's initial proposal and a significant quantity of new valuable information on the effectiveness of these methods in NPDES monitoring has become available, based on the use of these methods by many laboratories. The information on the success (or failure) of these methods in environmental laboratories is relevant to determining whether these methods should be promulgated at 40 CFR part 136. Therefore, EPA requests any relevant information on the performance of these methods.

Furthermore, the 10–18 proposal was published before enactment of the National Technology Transfer Advancement Act (NTTAA) of 1996. NTTAA requires EPA to consider standards developed by voluntary consensus standards bodies (VCSBs). EPA considers it appropriate to consider the VCSB methods described later in this preamble (many of which were revised after the 10–18 proposal), along with the other methods EPA is proposing to approve.

EPA intends to consider comments submitted on this proposal only when formulating the final rule. To the extent that anyone believes that comments submitted in response to the 10–18 proposal are still relevant, and wants EPA to consider them, such comments should be resubmitted in response to today's proposal.

a. Total Recoverable Elements Digestion

EPA proposes a broad-purpose digestion procedure (as described in EPA Method 200.2) for "total recoverable'' elements for use with:

- Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP–AES)
- Inductively Coupled Plasma-Mass Spectroscopy (ICP–MS)
- Stabilized Temperature Graphite Furnace Atomic Absorption Spectroscopy (STGFAA), and
- Flame Atomic Absorption Spectroscopy (FLAA) methods.

The total recoverable procedure uses a combination of nitric and hydrochloric acids (*aqua regia*) to prepare samples for analysis and is compatible with several measurement techniques. This generally allows laboratories to save some cost by reducing preparations and increasing flexibility in their choice of analytical techniques after digestion. The total recoverable digestion is less labor intensive than the approved (and equivalent) "total'' digestion method described in *Methods for Chemical Analysis of Water and Wastes* (MCAWW) "Metals (Atomic Absorption Methods)'' Section 4.1.3, while providing equivalent recovery of metals.

The total recoverable digestion procedure was incorporated into EPA Methods 200.7 (ICP–AES), 200.8 (ICP–MS) and 200.9 (STGFAA). It is published as the stand-alone Method 200.2, "Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements' (Rev. 2.8, 1994). EPA proposes total recoverable digestion for 200.7, 200.8, and 200.9 and allows the use of Method 200.2 as a digestion procedure in measuring some pollutants by FLAA, and VCSB-equivalents to EPA Methods 200.7, 200.8, and 200.9. However, Method 200.2 is not proposed for use with any standard GFAA methods due to the potential chloride interference. For GFAA methods, the total nitric acid digestion must be used.

The digestion procedure has been tested on various matrices using EPA Methods 200.7, 200.8 and 200.9 and has been found comparable to previously approved NPDES preparation procedures. Also, a joint EPA/AOAC International study of Method 200.8 provided further interlaboratory validation of the procedure.

b. Elements by ICP–MS

EPA proposes a multielement test procedure, Method 200.8 "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry'' (Revision 5.4, 1994) for the detection and quantification of 20 metals in aqueous wastewater samples. Method 200.8 has been used in the NPDES program through ATP approvals, and has been used in the NPDWR program for many years.

Method 200.8 determines elements using ICP–MS. Sample material in solution is introduced by pneumatic nebulization into a radio-frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are registered by an electron multiplier or Faraday detector, and the ion information is processed by a data handling system. Interferences relating to the technique are to be identified, and the results corrected accordingly. Such corrections must compensate for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift, as well as suppressions or enhancements of instrument response caused by the sample matrix, are to be corrected by using internal standards.

EPA developed ICP–MS Method 200.8 under a contract and in cooperation with AOAC International, and conducted a joint interlaboratory validation study of the method. The method description includes a list of the elements to which the method applies, sample collection practices, recommended analytical conditions, quality control practices, instrumental and method detection limits, and performance criteria based on the interlaboratory study data.

EPA also is proposing to approve VCSB methods that are equivalent to EPA Method 200.8: AOAC Method 993.14 [16th Edition], and ASTM Method D5673–02. These methods should provide performance similar to that obtained with Method 200.8.

c. Elements by STGFAA

EPA proposes a new multi-element test procedure, Method 200.9 “Determination of Trace Metals by Stabilized Temperature Graphite Furnace Atomic Absorption’’ (Revision 2.2, 1994) for the detection and quantification of 16 metals in aqueous wastewater samples. Method 200.9 has been used in the NPDES program through ATP approvals, and it has been used in the NPDWR program for many years.

Method 200.9 determines elements by stabilized temperature graphite furnace atomic absorption spectroscopy (STGFAA). In STGFAA, the sample and required matrix modifier are first pipetted onto the platform or a device which provides delayed atomization. The sample is then dried at a relatively low temperature (120 °C) to avoid spattering. Once dried, the sample is normally pretreated in a char or ashing step which is designed to minimize the interference effects caused by the sample matrix. After the char step, the furnace is allowed to cool prior to atomization. The atomization cycle is characterized by rapid heating of the furnace to a temperature where the metal (analyte) is atomized from the pyrolytic graphite surface. The resulting atomic cloud absorbs the element specific atomic emission produced by a hollow cathode lamp or a electrodeless discharge lamp.

Because the resulting absorbance usually has a nonspecific component (*e.g.*, black body radiation) associated with the actual analyte absorbance, an instrumental background correction device is necessary to subtract from the nonspecific component from the total signal. In the absence of interferences, the background corrected absorbance is directly related to the concentration of the analyte. Interferences relating to suppression or enhancement of instrument response caused by the sample matrix, is to be corrected by the method of standard addition.

The method description includes sample collection practices, recommended analytical conditions, quality control practices, method detection limits, and performance. Single laboratory studies show that Method 200.9 achieves performance comparable to ICP–AES and ICP–MS methods. In addition, Method 200.9 can achieve lower detection levels than ICP–AES methods (for all analytes in common between the methods), and ICP–MS methods (for certain analytes).

d. Hexavalent Chromium by Ion Chromatography

EPA proposes Method 218.6 “Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluent by Ion Chromatography’’ (Revision 3.3, 1994) for determination of hexavalent chromium. The NPDES program has used Method 218.6 through interim approvals.

Method 218.6 uses ion chromatography (IC) to determine hexavalent chromium (Cr(VI)) in samples. An aqueous sample is filtered through a 0.45 μm filter, and the filtrate is adjusted to a pH of 9 to 9.5 with a buffer solution. A measured volume of sample (50–250 μL) is introduced into the ion chromatograph. A guard column is employed to remove organics from the sample prior to separation of Cr(VI) as CrO_4^{2-}on an anion exchange separator column. Cr(VI) is determined by post column derivatization with diphenylcarbazide and passing through a low-volume flow-through cell for detection of the colored complex with a visible lamp detector at 530 nm.

Cooperating with ASTM Committee D–19 on Water, EPA conducted an interlaboratory validation study of EPA Method 218.6. The method description includes sample collection practices, recommended analytical conditions, quality control practices, method detection limits for Cr(VI), and performance criteria. The Method MDL in reagent water was 0.4 μg/L, twenty-five times lower than the DL for currently approved EPA Method 218.4, and performance was comparable to the currently approved method. For example, according to regression equations generated with data from the multilaboratory validation study, analyses of a 50 μg/L fortified reagent water sample would produce an average recovery of 103% and a relative standard deviation (RSD) of 5%.

ASTM, Standard Methods, and AOAC–International have approved this method as a standard test method under their consensus systems and have published it in their manuals of methods as follows: ASTM Method D5257–97, Standard Methods Method 3500–Cr C [20th Edition] and 3500–Cr E [18th, 19th], and AOAC Method 993.23 [16th Edition]. All three of these methods were derived from EPA Method 218.6 and are being proposed for approval.

e. Anions by Ion Chromatography

EPA proposes Method 300.0 “The Determination of Inorganic Anions in Water by Ion Chromatography,’’ (Revision 2.1, August 1993) for determination of common anions—bromide, chloride, fluoride, nitrate-N, nitrite-N, ortho-phosphate, and sulfate—in wastewater. Method 300.0 has been used for many years in the NPDWR program and in the NPDES program through interim approvals.

EPA Method 300.0 measures common anions using ion chromatography. A water sample is injected into a stream of carbonate-bicarbonate eluent and passed through a series of ion exchangers. Anions are separated on the basis of their relative affinities for a low capacity, strongly basic anion exchanger (guard and separator columns). The separated anions are directed through a hollow fiber cation exchanger membrane (fiber suppressor) or micromembrane suppressor bathed in continuously flowing strong acid solution (regenerant solution). In the suppressor, the separated anions are converted to their highly conductive acid forms, and the carbonate-bicarbonate eluent is converted to weakly conductive carbonic acid. The separated anions in their acid forms are measured by conductivity. They are identified on the basis of retention time as compared to reference standards. Quantitation is by measurement of peak area or peak height.

Cooperating with ASTM Committee D–19 on Water, EPA conducted an interlaboratory validation study of EPA method 300.0. The method includes results of the study, sample collection practices, recommended analytical conditions, quality control practices and estimated detection limits for the applicable analytes, and performance criteria. The method MDLs are lower than currently approved colorimetric methods, and performance was comparable to currently approved methods, with recovery falling within the 90–110% range and precision surpassing 10% RSD for all analytes in the working range of the method (mid-point of the calibrated range).

ASTM, Standard Methods, and AOAC-International approved and published the method under their consensus systems. EPA proposes approval of these following equivalents to EPA Method 300.0: ASTM Method D4327–97 and –03, Standard Method 4110 B [18th, 19th and 20th Ed.], and AOAC Method 993.30. EPA also is proposing EPA Method 300.1 "Determination of Inorganic Anions in Drinking Water by Ion Chromatography,'' now approved for NPDWR compliance monitoring, and which falls within the inherent flexibility (*i.e.,* is equivalent to) Method 300.0. This will further consistency among EPA monitoring programs.

f. Nitrate and Nitrite by Colorimetry

EPA proposes the use of automated and manual cadmium reduction methods for the determination of nitrate and nitrite, singly. Specifically, EPA proposes that EPA Methods 353.2, Standard Methods 4500–NO_3–E and F [18th, 19th, 20th] and 4500–NO_3–E and F (2000), ASTM Methods D3867–99(A) and (B), and I–4545–85 be used to determine nitrate and nitrite singly, as well as in combination, in NPDES compliance monitoring. Using these methods, "nitrate+nitrite'' can be determined by passing the sample through a cadmium reduction column (converting nitrate to nitrite for final analysis), and that the column can be by-passed to determine nitrite singly. With both of these values, nitrate can be determined by subtracting "nitrite'' from "nitrate+nitrite.'' This proposal is consistent with NPDWRs that allow cadmium reduction-based methods for nitrate+nitrite to measure nitrate and nitrite singly (see 40 CFR 141.23).

With regard to performance of automated methods, multi-laboratory data for EPA Method 353.2 indicates that analysis of a 1 mg/L nitrate sample will provide an average recovery of 100%, and a relative standard deviation (RSD) of 5.4%. Manual methods provide similar performance, with 4500–NO_3–E demonstrating an average recovery of 100% and RSD of 1% in single laboratory studies at concentrations near 1 mg/L. The equivalent versions of these methods published by other organizations should provide equivalent performance, given that they employ the same chemistry and procedures.

g. Chlorine by Low Level Amperometry

EPA proposes Standard Method 4500–Cl E [18th Ed.] and proposes 4500–Cl E [19th and 20th Ed.] and 4500–Cl E (2000) for the detection and quantification of low levels of chlorine in water (all editions are essentially the same). Method 4500–Cl E is a minor modification of the approved amperometric Method 4500–Cl D and can measure down to 10 µg/L chlorine. Federal and state permitting authorities requested such a method so they can assess compliance with effluent limits based on EPA and state water quality criteria for chlorine. You can find supporting performance data for the method at Journal of the Water Pollution Control Federation, Vol. 51, pages 2636–2640 (1979), a copy of which is included in the docket for this proposal.

h. Updated Versions of Currently Approved EPA Methods

In 1993 and 1994, EPA updated a number of methods from the "Methods for the Chemical Analysis of Water and Wastes'' (MCAWW) manual, and Method 200.7 (printed at 40 CFR part 136, Appendix A). For the most part, these updates were technically equivalent to previously approved versions, but offer the advantages of a consistent Environmental Monitoring Management Council (EMMC—an EPA committee consisting of EPA managers and scientists) format and explicit QC requirements which should result in improved data quality. Many of the versions are approved for NPDWR monitoring, so approval of these methods will further the goal of consistency among EPA monitoring programs. Finally many of these methods explicitly allow performance-based modifications, thereby increasing method flexibility.

All these methods, listed in Table I, were included in the 10–18 proposal. EPA proposes the approval of these additions and withdrawal of the old MCAWW versions.

TABLE I.—UPDATED REVISIONS PROPOSED IN 10–18–95

Parameter	Updated revisions		Currently approved revision (to be withdrawn)	
	Method	Revision	Method	Revision
Turbidity	180.1	Revision 2.0, August 1993*	180.1	1978
Multiple Metals	200.7	Revision 4.4, 1994*	200.7	1990
Mercury	245.1	Revision 3.0, 1994*	245.1	1974
Total Cyanide†	335.4	Revision 1.0 August 1993*	335.3	1978
Ammonia	350.1	Revision 2.0, August 1993	350.1	1978
TKN	351.2	Revision 2.0, August 1993	351.2	1978
Nitrate-Nitrite	353.2	Revision 2.0, August 1993*	353.2	1978
Phosphorus (all forms)	365.1	Revision 2.0, August 1993*	365.1	1978
Sulfate	375.2	Revision 2.0, August 1993*	375.2	1978
COD	410.4	Revision 2.0, August 1993	410.4	1978
Phenols	420.4	Revision 1.0, August 1993	420.2	1974

* Currently approved for use in NPDWR or NSDWR monitoring

† **Note:** EPA Method 335.4 is technically equivalent to the currently approved version of Method 335.3 when Method 335.3 is run in compliance 40 CFR 136.3, Table IB—Note 20 (specifically requiring the manual digestion of cyanide samples; if compared method-to-method, the procedures are quite different). However, as currently written, the sulfide removal procedure in Method 335.4 could lead to removal of particulate cyanide from the sample prior to analysis. Therefore, EPA proposes to add a footnote to the table to clarify the proper procedure for removing sulfide interferences. The footnote will require analysts to reconstitute samples treated for sulfide so that particulates are distilled along with the liquid sample.

Because these new versions of methods contain QC requirements (not previously included), and detection limits may have changed, EPA is particularly interested in comments regarding the ability of laboratories to achieve the specified QC requirements and detection limits.

EPA also requests comments on any additional costs that laboratories expect they might incur to comply with the QC requirements specified in the methods. EPA believes that many laboratories are already using thorough QC programs to ensure the reliability of the results they report, particularly for those methods already approved for NPDWR or NSDWR monitoring. EPA expects that any additional costs will be at least partially offset by the increased flexibility in these revisions (which explicitly allow analysts to reduce costs by introducing cost-effective innovations).

4. New EPA Methods

a. Mercury by CVAFS

EPA proposes Method 245.7, “Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry’’ [December 2003] (EPA–821–D–03–001) for measuring mercury (Hg) in wastewater. Method 245.7 provides reliable measurements of mercury at EPA water quality criteria levels. The method employs cold-vapor atomic fluorescence spectrometry (CVAFS), a brominating digestion creating minimal interference, and ultra-pure argon as the carrier gas. Samples are oxidized by a potassium bromate/potassium bromide reagent, sequentially pre-reduced with NH_2OHCl to destroy excess bromine, and the ionic mercury reduced with $SnCl_2$ to convert Hg(II) to volatile Hg(0). Hg(0) is then separated from solution by purging with high purity argon gas through a semi-permeable dryer tube. Once the Hg(0) passes into the inert argon gas stream, it is carried into the CVAFS detector cell to determine mercury concentration.

Method 245.7 is similar to EPA Method 1631 “Mercury in Water by Oxidation, Purge and Trap, and CVAFS,’’ originally promulgated on June 8, 1999 (64 FR 30434), for the NPDES program. Both methods use a CVAFS detector to measure low levels of mercury. Method 245.7 uses a liquid-gas separation and a dryer tube for analyte isolation, while Method 1631 uses a purge and gold trap isolation procedure. This difference makes Method 245.7 a low cost alternative to Method 1631 for measurement of trace-level mercury using CVAFS technology. For that reason, the Association of Metropolitan Sewerage Agencies (AMSA) petitioned the Agency to approve this alternative method. In response, EPA conducted a multi-laboratory validation of this method in 2001 to assess the method’s performance.

During this validation study, the method was tested on a variety of matrix types. In reagent water analyses performed in eight laboratories, average recoveries range between 85% to 105%, and relative standard deviations (RSDs) were below 15%. Percent recoveries in matrix spike/matrix spike duplicate industrial and municipal effluent samples ranged from 64% to 120%. The reported method detection limit is 5.0 ng/L based on the data from the eight participating laboratories. Following this study, Method 245.7 was updated to include the resulting performance criteria and to conform with recent EPA guidelines regarding contamination control.

Method 245.7 is available at *http://www.epa.gov/waterscience/methods/.* In addition, copies of the method and of the interlaboratory validation study report are in the docket supporting this proposal.

In addition to proposing EPA Method 245.7, EPA is soliciting comment on requirements for sample collection when using this method. On October 29, 2002, EPA promulgated a footnote at 40 CFR part 136 that includes sample preservation and storage requirements for samples collected for the determination of mercury using EPA Method 1631. This footnote states that: “Samples collected for the determination of trace level mercury (100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipment, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that have been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.’’ EPA is requesting comments and data demonstrating whether this footnote should or should not also be applied to Method 245.7.

5. New Voluntary Consensus Standard Bodies (VCSB) Methods

VCSB organizations asked EPA to approve several new VCSB methods for NPDES monitoring. In response to these requests, EPA proposes approval of the following VCSB methods. Some of the methods proposed are used in EPA monitoring programs today and/or have been approved as limited-use alternate test procedures.

a. Available Cyanide by Ligand Exchange-FIA

ASTM Method D6888–03 determines available cyanide (equivalent to “cyanide amenable to chlorination’’) using ligand exchange followed by flow injection analysis using gas diffusion separation and amperometric detection. It is very similar to the currently approved Method OIA–1677 (promulgated on December 30, 1999, 64 FR 73414).

Method D6888–03 was validated in an extensive intralaboratory study using several natural and industrial water matrices, and a 10-laboratory study using synthetic wastewater. Recoveries of potassium nickel cyanide and mercury (II) cyanide, the two strongest available cyanide complexes, ranged from 89.9 to 99.6% and 82.9 to 99.3%, respectively (in samples fortified to 100 μg/L as CN−).

Method D6888–03 states that either nickel cyanide or mercury (II) cyanide may be used to prepare quality control samples. However, for the purposes of NPDES compliance monitoring, EPA is proposing that only mercury (II) cyanide be used. Mercury (II) cyanide is a stronger complex than nickel cyanide (as evidenced by the slightly lower recoveries cited above), and, therefore, provides the most rigorous test for method performance. Currently approved Method OIA–1677 also specifies use of mercury (II) cyanide for the same reason.

In addition, two issues have come to EPA’s attention regarding the use of ligand exchange-amperometric detection methods for available cyanide. EPA seeks comment on both of these issues. First, EPA has received information suggesting that sulfide at levels below those detected with the lead acetate paper may produce false positive signals on the amperometric detection systems used in D6888–03 and OIA–1677 (see Zheng *et al.* “Evaluation and Testing of Analytical Methods for Cyanide Species in Municipal and Industrial and Contaminated Waters,’’ Environ. Sci. Technol. 2003, 37, 107–115). Lead acetate paper generally recommended for screening for the presence of sulfide interferences in cyanide methods, but the paper will not detect sulfides below approximately 5 ppm. For this reason, analysts suspecting a sulfide interference should test their sample with a more sensitive sulfide procedure and treat the sample accordingly. Appropriate test procedures for this purpose include the ion selective electrode (ISE) ASTM Method D4658–92(1996) and Standard Method 4500–S2–G which are proposed for use in today’s rule.

Second, EPA’s National Enforcement Investigation Center (NEIC) laboratory has found that when samples that contain significant solids are analyzed by OIA–1677, particles in the sample can settle out in the sample apparatus and also can clog the flow-injection system. As a result, measurements of cyanide in samples containing particulates decrease as the samples are allowed to stand in the sample tubes, and decrease as the system clogs. This decrease could be due to a number of factors, such as adsorption of released cyanide onto particulate or re-complexation of released cyanide with metals. This settling and clogging may be a problem in other similar cyanide-measurement systems that contain a rack of sample tubes, because the particles can settle during the time that the samples sit in these tubes. Suggested solutions to the problem are to (1) limit methods that use a rack of sample tubes to measurement of dissolved cyanide only; *i.e.*, samples that have been filtered through a 0.45-micron filter to remove particles, (2) to limit these methods to analysis of one sample at a time so that the settling cannot occur, (3) to limit the time between addition of the ligand-exchange reagents and the time of analysis to preclude settling, or (4) to require sample agitation during storage in the sample rack. EPA solicits comment on the problem, on the proposed solutions and on other possible solutions, and seeks data further characterizing the conditions under which the problem occurs and any solution(s) to the problem.

b. Cations by Ion Chromatography

ASTM International Standard Test Method D 6919–03, "Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography,'' applies to the simultaneous determination of dissolved inorganic alkali and alkaline earth cations and the ammonium cation in reagent water, drinking water, and wastewaters by suppressed and non-suppressed ion chromatography. While alkali and alkaline earth cations can be determined by alternative techniques such as AAS or ICP, ammonium cation in the same sample must be measured separately by a wet chemical technique such as colorimetry, titrimetry, or ammonia-selective electrode. Ion chromatography in a single automated run can determine ammonium plus all of the important inorganic cations including lithium, sodium, potassium, magnesium and calcium.

The cationic analytes are injected into a dilute acid eluent and separated by differential retention as they flow through guard and analytical columns packed with a low-capacity cation-exchange material. The separated cations are detected using conductivity detection, which is most sensitive when the background signal arising from the eluent has the lowest possible noise. One means to achieve low background noise is to combine the conductivity detector with a suppressor device that reduces the conductance of the eluent (*i.e.*, background noise) and also transforms the separated cations into their more conductive corresponding bases. Detection can also be achieved without chemical suppression, whereby the difference between the ionic conductance of the eluent and analyte cation is measured directly after the analytical column. This test method includes both suppressed and non-suppressed detection technologies.

A total of fourteen laboratories, employing one operator each, contributed data to the test method interlaboratory collaborative study. Three matrices were studied; reagent water, drinking water and wastewater. Each participant prepared and analyzed four Youden pairs for each of the six analytes in each of the three matrices. Analyte recoveries using Method D 6919–03 were determined in the range of 0.5–40 mg/L, with the specific concentration ranges tested for each cation varying slightly within this overall range. Method Detection Limits (MDLs) were confirmed in the 3–38 µg/L range. MDLs obtained by suppressed conductivity were approximately two times lower than the MDLs obtained by non-suppressed conductivity. The precision and recovery data for all analytes in all matrices tested are provided in the method. For example, across all analytes at concentrations of approximately 5 mg/L in drinking water, the range of multilaboratory recovery was 89–103% with relative standard deviation ranging from 4–15%. Quality control limits for the method and the data used to generate them are available in the docket supporting today's proposal.

Standard Test Method D 6919–03 is available from ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428–2959, United States. Douglas Later, the ASTM Subcommittee D19.05 Task Group Chairman for Method D 6919–03, can be reached at Dionex Corporation, 500 Mercury Drive, Sunnyvale, CA 94086. Telephone: (408) 481–4253, Fax: (408) 737–2470, e-mail: *Doug.Later@Dionex.com.* Copies of the method and the validation study report are in the docket for today's proposed rule.

c. Chloride by Potentiometry

Standard Method 4500–Cl–D [18th, 19th, and 20th Ed.] and (2000) is used to determine chloride in water by potentiometric titration, using a silver nitrate/solution with a glass and silver-silver chloride electrode system. During titration, an electronic voltmeter is used to detect the change in potential between the two electrodes. The end point of the titration is reached when the instrument reading at which the greatest change in voltage has occurred for a small and constant increment of silver nitrate added. The potentiometric method is a useful alternative to other approved methods when measuring chloride in colored or turbid samples that are not amenable to visual titration. The method also is included in NSDWRs, so its approval for NPDES program will further consistency between the wastewater and drinking water programs.

d. Chloride by Ion Selective Electrode

Method D512–89 (1999) C is a method for determining chloride ion in water by ion selective electrode (ISE). The stated range of the method is 2 to 1000 mg/L. Precision and bias were determined in reagent water and other matrices using a five-laboratory, seven-operator study. Recovery ranged from roughly 93–103% with RSD that generally fell within 5–10%. Additional data are available in the method.

e. Cyanide by Ion Selective Electrode

Standard Method 4500–CN–F [18th, 19th, and 20th Ed.] and (2000), and ASTM Method D2036–98 A, allow for analysis of cyanides, following distillation, using ion selective electrode (ISE) technology. These ISE methods have been used for a number of years in the context of NPDWR compliance monitoring, and have been approved in NPDES monitoring, on a limited-use basis, through the ATP program. Given the common use of these methods, and their ability to overcome certain interferences that could affect approved colorimetric methods, their inclusion at 40 CFR part 136 will be a useful addition to the suite of cyanide methods. Furthermore, the approvals will improve consistency across EPA programs.

ASTM conducted a six-operator, five-laboratory study of the ISE method as applied to reagent water and selected matrices. The effective range of the methods is 0.05–10 mg/L. Performance characteristics of the method are summarized in regression equations reproduced by both methods. As an example of performance, at 0.06 mg–

CN–/L reagent water, the interlaboratory percent recovery was 98% with a relative standard deviation of 14%. The Standard Method is technically identical and, therefore, should provide identical performance.

f. Sulfide by Ion Selective Electrode

Standard Method 4500–S2–G [18th, 19th and 20th Ed.] and ASTM Method D4658–92 (1996) determine sulfide in water using ion selective electrode (ISE). The Standard Method cites an applicable range of 0.032–100 mg/L with a 5% RSD at a concentration of 0.182 mg/L. The ASTM Method cites a range of 0.04–4000 mg/L, and a three-day, six-laboratory study demonstrated concentration variations of 6.5% (for 0.05–1.0 mg/L), 2.5% (1.0–100 mg/L), and 2.0% (100–4000 mg/L). Sulfide ISE methods are unaffected by sample color and turbidity and, therefore, provide a valuable substitute for approved colorimetric methods that may be affected by these interferences.

g. Nitrate by Ion Selective Electrode

Method 4500–NO_3–D [18th, 19th, 20th] and (2000) determines nitrate in water. The method employs an ion selective electrode (ISE) that develops a potential across a porous, inert membrane that holds in place a water-immiscible liquid ion exchanger. The method has a range of about 0.14 to 1400 mg nitrate-N/L, and a precision over the range of 2.5%. Nitrate ISE methods are unaffected by sample color and turbidity and therefore provide a valuable substitute for approved colorimetric methods that may be effected by these interferences. However, these ISE methods are susceptible to interferences due to variations in ionic strength. Therefore, when using these methods, analysts are to ensure that the sample and standard ionic strength match, or the method is to be performed in a way to prevent such interferences (*e.g.*, standard additions).

6. Updated Versions of Currently Approved Analytical Methods

a. EPA WET Methods

This rule proposes, and seeks comment on, an errata sheet for the following methods manuals:

- 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. (The "freshwater chronic manual'').
- 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. (The "freshwater acute manual'').

The errata sheet contains revisions to correct the following typographical errors:

1. Freshwater chronic manual page 274, section 1.10—

$$MSD = 2.36(0.097)[3\sqrt{(1/4)+(1/4)}] \; = \; 2.36(0.097)(\sqrt{2/4})$$

The "3'' before the square root symbol in the equation above should be removed.

2. Freshwater chronic manual page 274, section 1.11—The value 0.087 should be changed to 0.162.

3. Freshwater chronic manual page 10, section 4.4.1—"* * * The concentration of metals Al, As, Cr, Co, Cu, Fe, Pb, Ni, and Zn, expressed as total metal, should not exceed 1 µg/L each * * *''

4. Freshwater chronic manual page 11, section 4.8.3—"* * * The concentration of total organochlorine pesticides should not exceed 0.15 µg/g wet weight * * *''

5. Freshwater acute manual, section 7.2.3.1 Correct the concentration of selenium from 2 mg/L to 2 ug/L.

b. ASTM Methods

This rule proposes to approve a number of updated ASTM methods in NPDES compliance monitoring. Table II lists the most recently approved versions of the ASTM and the proposed versions for NPDES monitoring, as well as those proposed for NPDWR and NSDWR monitoring (proposed in Section IV.C.2). Copies of all the proposed methods are in the paper docket for review (they are not included in the e-docket due to copyright issues).

All of the proposed methods, except D512–89 (1999) (which is identical to the previous version), incorporate minor technical and/or editorial revisions that improve the methods. Previously approved versions of ASTM methods will remain approved.

TABLE II.—PROPOSED ASTM METHODS

Approved method	Proposed for wastewater	Proposed for drinking water	New method number
D511–93(A)	X	X	D511–03(A)
D511–93(B)	X	X	D511–03(B)
D512–89(A)	X		D512–89(99)(A)
D512–89(B)	X	X	D512–89(99)(B)
D516–90	X	X	D516–02
D858–95(A)	X		D858–02(A)
D858–95(B)	X		D858–02(B)
D858–95(C)	X		D858–02(C)
D859–94	X	X	D850–00
D888–92(A)	X		D888–03(A)
D888–92(B)	X		D888–03(B)
D1067–92	X		D1067–02
D1067–92(B)		X	D1067–02(B)
D1068–96(A)	X		D1068–03(A)
D1068–96(B)	X		D1068–03(B)
D1068–96(C)	X		D1068–03(C)
D1068–96(D)	X		D1068–03(D)
D1125–95(A)	X	X	D1125–95(99)(A)

TABLE II.—PROPOSED ASTM METHODS—Continued

Approved method	Proposed for wastewater	Proposed for drinking water	New method number
D1126–86(92)	X		D1126–02
D1179–93(A)	X		D1179–99(A)
D1179–93(B)	X	X	D1179–99(B)
D1246–95(C)	X		D1246–95(99)
D1252–95(A)	X		D1252–00(A)
D1252–95(B)	X		D1252–00(B)
D1253–86(92)	X	X	D1253–03
D1293–84(90)(A)	X		D1293–99(A)
D1293–84(90)(B)	X		D1293–99(B)
D1293–95		X	D1293–99
D1426–98(A)	X		D1426–03(A)
D1426–98(B)	X		D1426–03(B)
D1687–92(A)	X		D1687–02(A)
D1687–92(B)	X		D1687–02(B)
D1687–92(C)	X		D1687–02(C)
D1688–95(A)	X	X	D1688–02(A)
D1688–95(B)	X		D1688–02(B)
D1688–95(C)	X	X	D1688–02(C)
D1691–95(A)	X		D1691–02(A)
D1691–95(B)	X		D1691–02(B)
D1886–90(A)	X		D1886–94(98)(A)
D1886–90(B)	X		D1886–94(98)(B)
D1886–90(C)	X		D1886–94(98)(C)
D1889–94	X		D1889–00
D1890–90	X		D1890–96
D1943–90	X		D1943–96
D2330–88	X		D2330–02
D2460–90	X	Already Approved	D2460–97
D2972–97(A)	X		D2972–03(A)
D2972–97(B)	X	X	D2972–03(B)
D2972–97(C)	X	X	D2972–03(B)
D3086–90	X		D5812–96 (2002)
D3223–97		X	D3223–02
D3373–93	X		D3373–03
D3454–91	X	Already Approved	D3454–97
D3557–95(A)	X		D3557–02(A)
D3557–95(B)	X		D3557–02(B)
D3557–95(C)	X		D3557–02(C)
D3557–95(D)	X		D3557–02(D)
D3558–94(A)	X		D3558–03(A)
D3558–94(B)	X		D3558–03(B)
D3558–94(C)	X		D3558–03(C)
D3559–96(A)	X		D3559–03(A)
D3559–96(B)	X		D3559–03(B)
D3559–96(C)	X		D3559–03(C)
D3559–96(D)	X	X	D3559–03(D)
D3590–89(A)	X		D3590–02(A)
D3590–89(B)	X		D3590–02(B)
D3645–93(88)(A)	X		D3645–03(A)
D3645–93(88)(B)	X		D3645–03(B)
D3645–97(B)		X	D3645–03(B)
D3649–91		X	D3649–98a
D3697–92		X	D3697–02
D3859–98(A)	X	X	D3859–03(A)
D3859–98(B)	X	X	D3859–03(B)
D3867–90(B)	Already Approved	X	D3867–99(B)
D3972–97		X	D3972–02
D4107–91		X	D4107–98 (2002)
D4190–94	X		D4190–99
D4327–97	Proposed Today	X	D4327–03
D4382–95	X		D4382–02
D4657–92	X		D4657–92 (1999)
D4785–93	X	X	D4785–00a
D5174–97	X	X	D5174–02
D5317–93		X	D5317–98 (2003)

c. Standard Methods

This rule proposes to approve a number of updated Standard Methods in NPDES compliance monitoring; methods previously approved will continue to be applicable for compliance monitoring. Previously, USEPA has referenced approved Standard Methods using the edition of *Standard Methods for the Examination of Water and Wastewater* in which they were published. However, Standard Methods will now distribute methods on-line (likely in addition to printed volumes), so the option of only referencing an edition will not be practical.

For this reason, EPA will use a new numbering system to track the approved versions of Standard Methods. To indicate which version of the method is approved, the date of approval of a section by the Standard Method Committee will be used. For example, 2120 B–01 indicates the version of 2120 B approved by the Standard Methods Committee in 2001. The Committee Approval Date for a Standard Methods Section (*e.g.,* Section 2120) is provided in a footnote at the beginning of the Section.

Table III lists the most recently approved versions of the Standard Methods and the proposed versions for NPDES monitoring, as well as those proposed for NPDWR and NSDWR monitoring (proposed in Section IV.C.3). While a number of methods contain no changes from previously approved version, some incorporate minor technical and editorial revisions to improve user-friendliness, update references, and correct errors. Methods that were revised from previous versions are indicated on the table. Previously approved versions of Standard Methods will remain approved. Copies of all the proposed methods are in the paper docket for review (they are not included in e-docket due to copyright issues), see Section IV.C.2.

TABLE III.—PROPOSED STANDARD METHODS

Revised from standard methods [most recent approved version]	revised	proposed for wastewater	proposed for drinking water	New number
2120 B [20th]	X	X	X	2120 B–01
2130 B [20th]		X		2130 B–01
2150 B [20th]			X	2150 B–97
2310 B [20th]		X		2310 B–01
2320 B [20th]		X	X	2320 B–97
2340 B [20th]	X	X		2340 B–97
2510 B [20th]	X	X	X	2510 B–97
2540 B [20th]		X		2540 B–97
2540 C [20th]		X	X	2540 C–97
2540 D [20th]		X		2540 D–97
2540 F [20th]		X		2540 F–97
2550 B [20th] (listed as "2550" for drinking water regulations)	X	X	X	2550 B–00 (2550–00)
3111 B [19th]	X	X	X	3111 B–99
3111 C [19th]		X		3111 C–99
3111 D [19th]		X		3111 D–99
3112 B [19th]		X	X	3112 B–99
3113 B [19th]		X	X	3113 B–99
3114 B [19th]	X	X	X	3114 B–97
3120 B [20th]	X	X	X	3120 B–99
3500–Al B [20th]		X		3500–Al B–01
3500–As B [20th]	X	X		3500–As B–97
3500–Ca B [20th]		X	X	3500–Ca B–97
3500–Cr B [20th]	X	X		3500–Cr B–01
3500–Cu B [20th]	X	X		3500–Cu B–99
3500–Cu C [20th]	X	X		
3500–Fe B [20th]	X	X		3500–Fe B–97
3500–Pb B [20th]		X		3500–Pb B–97
3500–Mg B [20th]			X	3500–Mg B–97
3500–Mn B [20th]		X	X	3500–Mn B–99
3500–K B [20th]		X		3500–K B–99
3500–Na B [20th]	X	X		3500–Na B–97
3500–V B [20th]		X		3500–V B–97
3500–Zn B [20th]		X		3500Zn B–97
4110 B [20th] (proposed for NPDES in this rule)		X	X	4110 B–00
4500–B B [20th]		X		4500–B B–00
4500–CN–D [20th]		X		4500–CN D–99
4500–CN–E [20th]		X	X	4500–CN E–99
4500–CN–F [20th] (proposed for NPDES in this rule)		X	X	4500–CN F
4500–CN–G [20th]		X	X	4500–CN G–99
4500–Cl B [20th]		X		4500–Cl B–00
4500–Cl C [20th]		X		4500–Cl C–00
4500–Cl D [20th]		X	X	4500–Cl D–00
4500–Cl E [20th] (proposed for NPDES in this rule)		X	X	4500–Cl E–00
4500–Cl F [20th]		X	X	4500–Cl F–00
4500–Cl G [20th]		X	X	4500–Cl G–00
4500–Cl H [20th]			X	4500–Cl H–00
4500–Cl I [20th]			X	4500–Cl I–00
4500–Cl–B [20th]		X		4500–Cl–B–97
4500–Cl–C [20th]		X	X	4500–Cl–C–97

TABLE III.—PROPOSED STANDARD METHODS—Continued

Revised from standard methods [most recent approved version]	revised	proposed for wastewater	proposed for drinking water	New number
4500–Cl-E [20th]		X		4500–Cl-E–97
4500–ClO$_2$ C [20th]			X	4500–ClO C–00
4500–ClO$_2$ E [20th]		X	X	4500–ClO2
4500-F-B [20th]		X	X	4500-F-B–97
4500-F-C [20th]		X	X	4500-F-C–97
4500-F-D [20th]		X	X	4500-F-D–97
4500-F-E [20th]		X		4500-F-E–97
4500-H+ B [20th]	X	X	X	4500-H+ B–00
4500-NH$_3$ B [20th]		X		4500-NH$_3$ B–97
4500-NH$_3$ C [20th]		X		4500-NH$_2$-C–97
4500-NH$_3$ D [20th]		X		4500-NH$_3$ D–97
4500-NH$_3$ E [20th]		X		4500–NH$_3$ E–97
4500-NH$_3$ G [20th]	X	X		4500-NH$_3$ G–97
4500-NO$_2$ B [20th]		X	X	4500-NO$_2$ B–00
4500-NO$_3$-D [20th]		X		4500-NO$_3$- D–00
4500-NO$_3$-E [20th]	X		X	4500-NO$_3$- E–00
4500-NO$_3$-F [20th]		X	X	4500-NO$_3$ F–00
4500-NO$_3$-H [20th]		X		4500-NO$_3$- H–00
4500-N$_{org}$ B [20th]		X		4500-N$_{org}$ B–97
4500-N$_{org}$ C [20th]		X		4500-N$_{org}$ C–97
4500-O C [20th]		X		4500-O C–01
4500-O G [20th]		X		4500-O G–01
4500-O$_3$ B [19th] (4500-O$_3$ B [20th] is proposed in this rule)	X		X	4500-O$_3$ B–97
4500-SiO$_2$ C [20th] is proposed in this rule)	X		X	4500-SiO$_2$> C-97
4500-SiO$_2$ C [20th]	X	X		4500-SiO$_2$ C–97
4500-SiO$_2$ D [20th]			X	4500-SiO$_2$ D–97
4500-SiO$_2$ E [20th]			X	4500-SiO$_2$ E–97
4500-SiO$_2$ F [20th]			X	4500-SiO$_{2\_}$ F–97
4500-S^{2-} D [20th]		X		4500-S^{2-} D–00
4500-S^2 F [20th]		X		4500-S^{2-} F–00
4500-S^{2-} G [20th]		X		4500-S^{2-} G–00
4500-SO$_3$3^{2-} B [20th]		X		4500-SO$_3$3^{2-} B–00
5210 B [20th]	X	X		5210 B–01
5220 C [20th]		X		5220 C–97
5220 D [20th]		X		5220 D–97
5310 B [20th]		X		5310 B–00
5310 C [20th]		X		5310 C–00
5310 D [20th]		X		5310 D–00
5520 B [20th]	X	X		5520 B–01
5540 C [20th]	X	X	X	5540 C–00
6200 B [20th]	X	X		6200 B–97
6200 C [20th]		X		6200 C–97
6410 B [20th]		X		6410 B–00
6420 B [20th]	X	X		6420 B–00
7110 B [20th]	X	X	X	7110 B–00
7110 C [20th]			X	7110 C–00
7120 [20th]	X		X	7120–97
7500-Cs B [20th]			X	7500-Cs B–02
7500-I B [20th]			X	7500-I B–00
7500-I C [20th]			X	7500-I C–00
7500-I D [20th]			X	7500-I D–00
7500-Ra B [20th]		X		7500-Ra B–01
7500-Ra C [20th]		X	X	7500-Ra C–01
7500-Ra D [20th]			X	7500-Ra D–01
7500-Sr B			X	7500-Sr B–01
7500–^{3}H B [20th]	X		X	7500–^{3}H B–00
7500-U B [20th]			X	7500-U B–00
7500-U C [20th]			X	7500-U C–00
9215 B [20th]			X	9215 B–00
9221 A [20th]			X	9221 A–99
9221 B [20th]		X	X	9221 B–99
9221 C [20th]	X	X	X	9221 C–99
9221 D [20th]		X	X	9221 D–99
9221 E [20th]		X	X	9221 E–99
9222 A [20th]	X		X	9222A–97
9222 B [20th]	X	X		9222 B–97
9222 C [20th]			X	9222 C–97
9222 D [20th]		X	X	9222 D–97
9223 [20th]			X	9223–97
9230 B [20th]	X	X		9230 B–93

TABLE III.—PROPOSED STANDARD METHODS—Continued

Revised from standard methods [most recent approved version]	revised	proposed for wastewater	proposed for drinking water	New number
9230 C [20th]		X		9230 C–93

d. AOAC International

This rule proposes to update references to approved methods from AOAC International to include the versions of those methods published in the 16th edition of Official Methods of Analysis of AOAC International, 1995, for use in NPDES compliance monitoring. Approved AOAC methods from earlier editions of Official Methods of Analysis of AOAC International will continue to be applicable for compliance monitoring.

7. Method Modifications, Analytical Requirements, and Reporting Requirements

a. Replacement of Mercury Catalyst in TKN Methods

Mercuric sulfate is used as a catalyst in some approved methods for determining total Kjeldahl nitrogen (TKN). Mercuric sulfate is a toxic hazard and the presence of mercury in used reagents increases waste disposal costs. For these reasons, EPA proposes to explicitly require the substitution of copper sulfate for mercuric sulfate in all TKN methods. Copper sulfate exhibits significantly less toxicity than mercuric sulfate. The European community has already eliminated mercuric chloride from their total nitrogen methods in favor of less toxic catalysts, and some approved methods (*e.g.*, 19th and 20th Ed. Standard Methods for TKN) have even included copper sulfate explicitly as a catalyst (evidencing the technical acceptability of the substitution).

b. Approval of Additional Standards for Turbidity

EPA is proposing to formally approve the use of styrene divinylbenzene beads (AMCO–AEPA–1 Standard) and Hach StablCal as alternatives to the presently approved formazin standard. Formazin is prepared using hydrazine sulfate, a known carcinogen. The approval of AMCO–AEPA–1 and Hach StablCal would eliminate the need to handle hydrazine sulfate, and would, therefore, improve laboratory safety. The NPDES ATP program has recognized AMCO–AEPA–1 Standard (listed in EPA Method 180.1 as an approved primary standard for drinking water) and Hach StablCal as acceptable alternatives to formazin for a number of years. Inclusion at 40 CFR 136 would formalize this acceptance nationwide.

c. Use of Capillary Columns

EPA proposes to allow the use of capillary (open tubular) GC columns with EPA Methods 601–613, 624, 625, and 1624B provided that all quality control (QC) tests in these methods are performed and all QC acceptance criteria are met. This action would codify EPA's general practice of allowing capillary GC columns in placed of the packed columns described in the above methods. However, when employing capillary columns, the retention times of analytes can change substantially. Therefore, EPA proposes to require that analysts prepare analyte retention time tables based on the capillary columns that they used.

d. Analytical Requirements for Multi-analyte Methods (Target Analytes)

EPA proposes to clarify that analysts need only meet method performance requirements for target analytes (those analytes being measured for NPDES reporting). Some analysts interpreted performance requirements in methods to mean that requirements for every analyte in a method must be met. However, attempting to meet the performance requirements of non-target analytes can add substantial cost (due to extra analyses, extra preparation of standards, etc.) with little or no benefit to the quality of target analyte data.

e. Requirements for Approval of Method Modifications

EPA also proposes codification of method flexibility provisions and analytical requirements at 40 CFR 136.6. This new part describes potentially allowable method modifications and requirements that analysts would need to meet to use these modifications without prior EPA approval. The part would also clarify the analytical requirements for multi-analyte methods, and codify EPA's allowance of capillary columns with gas chromatography methods that currently specify the use of packed columns.

In order to evaluate method modifications, the analyst would be required to assess performance by analyzing test samples and comparing the results with performance benchmarks for the unmodified method. The quality control (QC) tests and QC acceptance criteria provided in many of the approved methods generally would serve this purpose. At a minimum, the analyst would need to evaluate performance in wastewater matrices and include both initial (start-up) and ongoing procedures to evaluate performance. If the tests and criteria in a method meet these minimum standards, they would be used to evaluate a modification. If the tests and criteria in a method do not meet these minimum standards, analysts would use QC tests and acceptance criteria specified in *Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA–821–B–98–002; March 1999) (ATP Protocol). The applicable tests, which are common to the analytical community (*e.g.*, calibration verification tests, matrix spike-matrix spike duplicate tests), are described in Section 3.5 of the ATP Protocol. QC acceptance criteria for these tests are found in Table IF of the ATP Protocol.

When applying the ATP protocol, analysts would need to use the tests and criteria in initial validation and ongoing verification. The ongoing verification would include 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 of a discharge analyzed), and analysis of an ongoing precision and recovery sample and a method blank with each batch of 20 or fewer samples.

The actions proposed would codify past EPA policy that has been specified in certain approved methods and guidance. For example, with regard to allowable method modifications, the proposed allowance of an increase of sample volumes up to 25 mL for purge-and-trap methods, recognizes the existing flexibility in EPA Method 524.2 (and places reasonable limits on sample volumes based on the demonstrated performance of that method) and the use of salt in sample extraction recognizes recommendations from EPA's *Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring*. Increasing the sample volume has been used as one means to lower the detection limits of some purge-and-trap procedures and EPA recognizes that the purging efficiency of some of the analytes of interest may be adversely affected by the increased sample volume and may lead to decreased precision and recovery for those analytes. When using an increased sample volume, EPA strongly recommends the use of one or more surrogate analytes that are chemically similar to the analytes of interest. Use of these surrogates should assist in the identification of analytical errors that may result from the increased sample volume. EPA further recognizes that increasing the sample volume may necessitate changes to the configuration of the purge-and-trap device in order to provide a water column height of at least 5 cm in the purge vessel. EPA requests comments and data regarding whether the existing quality control procedures and the use of calibration procedures in which the standards that are also purged are sufficient to substantiate the performance of these methods when the sample volume is increased beyond 5 mL. EPA also requests comment as to whether the standards for evaluating modifications are adequately defined, and whether the potentially allowable modifications should be expanded, reduced, or changed.

Finally, EPA requests comment on the reference to the ATP protocol in 40 CFR 136.6. The proposed 40 CFR 136.6 only references the ATP protocol guidance to establish baseline QC tests and acceptance criteria for modifications made under 40 CFR 136.6 where such criteria are not available in methods. The reference to the ATP protocol would not, however, bind EPA to apply the ATP protocol as written to ATPs processed under 40 CFR 136.4 and 136.5. EPA may modify the ATP protocol guidance or apply different requirements for validation of ATPs under 40 CFR 136.4 and 136.5, as appropriate, without notice and opportunity for comment.

f. Clarification of Reporting Requirements

EPA proposes to add section 136.7 to clarify that a quality control (QC) failure does not grant relief of timely reporting of results to a regulated entity, and that results be reported to the level specified in the method or required in the permit, whichever is lower. EPA emphasizes that this clarification does not create any new or additional reporting requirements. In fact, the methods in this part do not create reporting requirements at all. Reporting requirements are created when a regulatory or control authority requires reporting of results upon use of a method at this part.

8. Withdrawal of Methods

a. Previous Versions of Updated Methods and Outdated Methods

EPA proposes to remove some older versions of EPA methods and replace them with updated versions, (see Table I). The updated versions include quality control procedures that should help improve data reliability. In addition, EPA is proposing deletion of most methods from EPA's Methods for the Chemical Analysis of Water and Wastes. In many cases, these methods were replaced with newer versions of the EPA methods, and in all cases approved alternatives (either published by EPA or VCSBs like ASTM and Standard Methods) are available.

b. Liquid-Liquid Extraction Methods for Dichlorobenzenes

EPA proposes to delete liquid-liquid extraction (LLE) methods, including EPA Methods 612 and 625 and Standard Methods Method 6410 B, as approved procedures for 1,2–dichlorobenzene, 1,3–dichlorobenzene, and 1,4-dichlorobenzene (originally included in the 10–18 proposal). While these compounds can be determined by these LLE methods, significant losses of these volatiles can occur using the prescribed sample collection procedures in the LLE methods, resulting in relatively low recovery of these compounds. These compounds are more accurately and precisely analyzed by EPA Method 624 or 1625B (an isotope-dilution method that compensates for evaporation losses).

c. CFC-based Oil and Grease Methods

EPA proposes to withdraw approval for all oil and grease methods that use chlorofluorocarbon-113 (CFC–113; Freon–113) as an extraction solvent because CFC–113 is a Class I ozone-depleting substance (ODS). On May 14, 1999 (64 FR 26315), EPA approved EPA Method 1664A as a replacement for Freon-based oil and grease methods to reduce dependency on CFC–113 (Method 1664A uses n-hexane as an extraction solvent). On March 13, 2001 (66 FR 14759), EPA published rules that would eliminate the global laboratory use exemption for ODSs produced or imported after December 31, 2001, for testing oil and grease and petroleum hydrocarbons in water; on November 1, 2001 (66 FR 55145), EPA proposed to codify this elimination. To further reduce reliance on ODSs, EPA proposes to withdraw EPA Method 413.1 and USGS Method I–4540–85 from use, and to specify that only n-hexane extraction solvent (as used in EPA Method 1664A) is approved with the remaining methods. The withdrawal and replacement would take effect on December 31, 2005, consistent with the November 1, 2001, proposal. This would allow those remaining dischargers and permittees who have not switched to n-hexane methods (*e.g.*, 1664A) time to become completely familiar with the alternative n-hexane methods.

B. Sample Collection, Preservation, and Holding Time Requirements for NPDES Compliance Monitoring and Pretreatment

1. Updates to Sampling Requirements at 40 CFR Parts 122, 136, and 403

This rule proposes to correct inconsistencies in sampling requirements at 40 CFR parts 122, 136, and 403. These inconsistencies were inadvertently created by past rulemakings. In addition to correcting the current language, references back to 40 CFR part 136 would be added to Sections 122 and 403 because the analytical methods and sampling requirements promulgated at 40 CFR part 136 often give detailed and up-to-date instructions on sample collection. Also recognizing that a single section of the CFR is the primary source for sample collection requirements will prevent future inconsistencies.

2. Revisions to 40 CFR Part 136, Table II

40 CFR part 136, Table II specifies sampling, preservation, and holding time requirements. This proposal would make a number of additions and modifications to these tables to reduce confusion and reflect current understanding of sample preservation requirements. The proposed changes are:

Changes to General Requirements

EPA proposes to clarify the abbreviation “do.'' (used extensively in Table II), and to change the general sample preservation temperature from 4 °C to ≤6.00 °C (unfrozen).

“Do.'' means “ditto''; *i.e.*, that the entry immediately above the “do.'' applies. This definition has always been the meaning of “do.,'' but EPA would add language to Table II to clarify this point.

EPA has received requests to make temperature requirements consistent with those of the National Environmental Laboratory Accreditation Committee (NELAC). NELAC has adopted a standard of 4 ± 2 °C for sample preservation temperature and has asked

EPA to adopt this definition. EPA has proposed a ≤6.00 °C (unfrozen) sample perservation temperature because maintenance of a 4 ± 2 °C temperature requires an active refrigeration system (which will raise sample shipping costs), and because EPA is not aware of any evidence to suggest that allowing refrigeration below 2 °C (the lower limit of NELAC standard) will adversely effect samples.

Because many approved methods list preservation temperatures, adopting the ≤6.00 °C (unfrozen) sample preservation temperature would cause inconsistencies between Table II and methods that list a 4 °C sample preservation temperature. Therefore, EPA proposes to add a note to Table II specifying that preservation temperatures in Table II supersede all temperature requirements listed in approved methods or other sources.

Requirements for Inorganic Parameters

EPA is proposing changes to parameter 10 (boron), 18 (hexavalent chromium), and 23–24 (cyanides), 25 (fluoride), 35 (mercury), and metals. Changes to boron and fluoride are proposed because of proposed changes in footnote 1 of Table II described later in this section, and make no substantive changes. For boron, EPA proposes to remove "PTFE'' because this information duplicates the allowed use of fluoropolymer in proposed footnote 1. Similarly, the entry for fluoride will explicitly limit sample collection to polyethylene containers.

For cyanides, EPA proposes to re-draft listings in Table II to include total cyanide, cyanide amenable to chlorination, and available cyanide (to be consistent with 40 CFR part 136, Table IB). This, too, does not pose a substantive change.

The other proposed changes are substantive. For hexavalent chromium, EPA proposes to increase the holding time for chromium 6 (CrVI) from 24 hr to 28 days when the sample is preserved to pH 9.3 to 9.7 using sodium hydroxide and the ammonium sulfate buffer solution specified in EPA Method 218.6. (Method 218.6 is also being proposed today.) EPA has received a presentation and spreadsheet from Montgomery-Watson Laboratories and East Bay MUD supporting the increase in holding time and has placed the presentation and spreadsheet in the Docket for today's proposal. EPA solicits further data supporting, refuting, or causing modification of the proposed increase in holding time.

For mercury, requirements would be divided by methodology (as each requires different sample handling and preservation techniques) and sample type, and tissue samples frozen to < − 10 °C could be held for 10 years under certain conditions. Finally, for metals (elements) other than boron, hexavalent chromium, and mercury, EPA proposes to allow sample preservation (in the original sample) with nitric acid 24 hours prior to analysis. In other words, acid preservation in the field for elements would not be required except for boron, hexavalent chromium, and mercury. This proposal reflects current EPA policy, prevents the shipment of dangerous acidic materials, and is supported by data showing that metals adsorbed to a sample container will resolubilize with 24 hours of acidification.

Requirements for Organics in Table IC

EPA proposes to split the entry for field preservation into separate entries for tissue and for solid and mixed phase samples, allowing a seven-day holding time for mixed phase samples, a 24–hour holding time for tissues in the field, and one-year holding time for all samples frozen to < − 10 °C. These changes reflect that tissue samples must be frozen within 24 hours to maintain sample integrity.

Footnotes In Table II

This rule proposes modifications to footnotes 1, 2, 4, 5, 6, 7, 11, and 13 to 40 CFR 136.3, Table II. By editing footnote 1, EPA would allow fluoropolymer sample containers for all tests that presently allow use of polyethylene, except fluoride (for which this container is not appropriate). This change reflects the common use of fluoropolymers (like PTFE) in the laboratory, and their value in making unreactive sample containers.

EPA proposes to revise footnote 2 to clarify current sampling requirements. Similarly, EPA proposes to revise footnote 4 to clarify that the start of holding times. The holding time for a grab sample starts at the time of sample collection. The holding time for a composite sample starts at the time the last grab sample component is collected.

EPA proposes to revise footnote 4 to clarify that for bacteriolgical samples, the holding time of six hours may followed by two hours to analyze the sample. EPA has received questions about whether the holding time of six hours includes sample analysis time or not.

EPA proposes to update footnotes 5 and 6 as part of revisions to the preservation requirements at 40 CFR 136.3, Table II to reflect the options included in certain proposed and currently approved methods. EPA has received complaints about current sample preservation techniques (such as the addition of ascorbic acid as an anti-chlorinating agent) and believes the procedures that EPA proposes would prove more successful at providing high quality data. By citing all the recommended preservation options in approved methods, EPA expects analysts to chose those that provide the most accurate results.

EPA also is considering alternative preservation and interference removal procedures for cyanide samples. In particular, for samples containing sulfides, EPA is proposing to allow use of bismuth (as opposed to cadmium or lead) or lowering the sample pH and stripping out hydrogen sulfide with air prior to addition of sodium hydroxide. Lead and cadmium may inadvertently promote the precipitation of metal-cyanide complexes, leading to the under-reporting of total cyanide. EPA requests comment on all the preservation procedures proposed and under consideration, as well as alternatives which could improve total cyanide recoveries. EPA further requests that pertinent data and references to relevant articles be included with such comments.

Footnote 7 would be revised to clarify that samples analyzed for dissolved metals should be filtered within 15 minutes of collection (currently the footnotes specifies that samples should be filtered "immediately''). Footnote 11 would be revised to reflect the proposed change in sample preservation temperature to ≤6.00 °C, described above. Also, footnote 13 would be revised to allow the storage of sample extracts for 30 days if stored at <0 °C (based on results of studies with EPA Method 553).

C. Editorial Revisions and Clarifications to 40 CFR Parts 122, 136, 455, and 465

This proposal would make many other minor changes to 40 CFR part 136. These changes are intended to clarify existing regulations, or increase method flexibility.

40 CFR Part 122

EPA is considering two options to clarify regulations regarding the use of analytical methods specific to Title 40 of the CFR, Chapter I, Subchapters N and O (effluent guidelines and sewage sludge, respectively). Currently, regulations at 40 CFR part 122 (that implement the general provisions of the NPDES regulations), state that NPDES monitoring must be conducted with methods specified at 40 CFR part 136. As a result, 40 CFR part 122 may confuse the reading of effluent guidelines regulations at 40 CFR parts 400–471 (Chapter I, Subchapter N), and with sewage sludge regulations at 40 CFR part 501–503 (Chapter I, Subchapter O), because methods are included in those regulations that are not specified at 40 CFR part 136. For example, the pulp, paper, and paperboard point source category (40 CFR part 430) provides two methods specifically for use in that category at 40 CFR part 430, Appendix A (EPA Methods 1650 and 1653). The intent of including these methods at 40 CFR part 430 was that permit writers would specify their use in permits covering the pulp, paper and paperboard effluent. However, the language at 40 CFR part 122 could be read to defeat this intent.

To harmonize the existing regulations, EPA is considering two options. Under Option 1, EPA may modify language at 40 CFR part 122 to explicitly allow use of methods at 40 CFR part 136 or that are specifically included in regulations that cover the discharge. For example, the following language in [brackets] would be added to 40 CFR 122.21(g)(7)(i):

> (7) *Effluent characteristics.* (i) Information on the discharge of pollutants specified in this paragraph (g)(7) (except information on storm water discharges which is to be provided as specified in § 122.26). When "quantitative data'' for a pollutant are required, the applicant must collect a sample of effluent and analyze it for the pollutant in accordance with analytical methods approved under part 136 of this chapter [unless a method is specified for an industry-specific waste stream at 40 CFR subchapters N or O]. When no analytical method is approved [under part 136 or specified under subchapters N or O,] the applicant may use any suitable method but must provide a description of the method.

Similar changes would be made to 40 CFR 122.1(a)(4), 122.41(j)(4), 122.41(l)(4)(ii), and 122.44(i)(1)(iv), as described in the regulatory text of this rule.

Under Option 2, EPA would add a table or tables to 40 CFR part 136 listing methods that are included in other parts of the CFR and the regulations to which they are applicable. This approach has been taken in the past with certain industry-specific effluent guidelines. For example, 40 CFR part 136, Table IF specifies methods that may be used at 40 CFR part 439 (pharmaceutical manufacturing point source category), and today's rule proposes the addition of Table IG to 40 CFR 136 to list methods for use at 40 CFR 455 (pesticide chemical point source category). EPA solicits comments on both approaches, or other options that may be preferable for resolving the current confusion.

40 CFR 136.3, 136.4 and 136.5

EPA proposes to revise all occurrences of "Director of the Environmental Monitoring Systems Laboratory'' and "Director, Analytical Methods Staff'' to "Alternate Test Procedure Program Coordinator, Washington, DC'' to reflect EPA's current ATP Program management. In addition, addresses for submission of ATPs will be updated to reflect the current location of the Alternate Test Procedure Program Coordinator.

40 CFR Part 136, Table IA

EPA proposes to delete footnote 4, which provides reference information for Standard Methods. Footnote 4 is not needed because the reference is recognized by the laboratory and regulated community, and reference information is provided at 40 CFR 136.3(b).

40 CFR Part 136, Table IB

EPA proposes minor edits to footnotes 1, 4, and 6. EPA proposes to add an NTIS order number to footnote 1, revise metals digestion requirements to footnote 4 (in light of changes previously in this Section), and to remove the word "company'' from footnote 6 (because entities that conduct testing are not always companies). Also, EPA proposes to revise the format of references to footnote 10 to be consistent with other sections of the CFR.

40 CFR Part 136, Table IC and ID

EPA proposes to remove the "Note'' regarding warning limits "interim'' status from footnote 7 to both tables, because these limits have been in use for more than 15 years without difficulties (beyond those always encountered when first starting to use a method).

40 CFR Part 136, Table IE

EPA proposes to add an NTIS reference number to footnote 1.

40 CFR Part 136, Table IG and 40 CFR 455

EPA proposes to move Table 7 from 40 CFR part 455, to 40 CFR part 136, Table IG. EPA proposes this change to further consolidate lists of analytical methods in a single section of the CFR.

Addition of 40 CFR 136.6

EPA proposes to add the additional method flexibility and analytical requirements discussed in Section III.A.7.

Addition of 40 CFR 136.7

EPA proposes to add the clarified reporting requirements discussed in Section III.A.7.

Changes to 40 CFR Part 465

This rule proposes to remove the exemption for Freon-based oil and grease methods (described *supra*). The Coil Coating Point Source Category at 465.03 contains a method for determination of petroleum hydrocarbons using a freon extraction method. EPA proposes to remove this method and to replace it with a reference to EPA Method 1664A for determination of non-polar materials (NPM), which is generally equivalent to total petroleum hydrocarbons. EPA has received many requests to allow the use of Method 1664A for this industrial category. This change will further the goal of reducing the use of ozone depleting substances.

IV. Summary of Proposed Revisions to Drinking Water Regulations

A. Vendor Developed Methods

1. Anions by CIE–UV

Waters Corporation CIE/UV Method (D6508, Rev. 2), described in Section III.A.1.a above, is a new method that employs capillary ion electrophoresis to determine common anions in wastewater and drinking water. This method is being proposed today for use in NPDWR and NSDWR compliance monitoring for determination of the common anions.

2. Free Chlorine by Color Comparison Test Strip

This rule proposes to allow States the option of approving ITS free chlorine test strips as a test kit for the measurement of free chlorine. The ITS test strip is configured with a "color pad'' attached to a plastic holder. The color pad contains 3,3,5',5'-tetramethylbenzidine (TMB) which reacts with chlorine to produce a color change that is proportional to the amount of free chlorine in the sample. The chlorine concentration is quantified by comparison of this color with an ITS color chart.

The use of ITS free chlorine test strips has been discussed in literature and has been validated in drinking water using two interlaboratory validation studies. The studies were performed to characterize the false negative and false positive rates of the strips, the precision and recovery using the strips, the sensitivity of the strips, and the variability of test strips between lots. To eliminate potential analyst bias, all studies were double-blind and random.

The false positive and false negative rates were 0–1%. Method precision and recovery was characterized in multiple matrices at multiple concentrations. For example, free chlorine recovery was approximately 100%, and relative standard deviation (RSD) was generally below 20% for analysis of drinking water samples fortified with 0.1 ppm of free chlorine. Method sensitivity was demonstrated to be sufficient for monitoring chlorination levels at 0.1 ppm or above; chlorination levels required by NPDWRs is 0.2 ppm. Finally, results did not appear to vary across different lots of ITS strips.

The use of the test strips is described in Method D99–003, "Free Chlorine Species'' (HOCl- and OCl-) by Test Strip'' [Revision 3.0, November 21, 2003]. A copy of Method D99–003 and the method validation study report are in the docket supporting this rule. In addition, copies of Method D99–003 and test strips are available from Industrial Test Systems, Inc., 1875 Langston St., Rock Hill, SC 29730.

3. Available Cyanide by Ligand Exchange—FIA

This rule proposes approval of two similar methods for the determination of available cyanide: Method OIA–1677, DW and ASTM D6888–03. Studies have shown that available cyanide is equivalent to cyanide amenable to chlorination (CATC), and, therefore, that available cyanide methods can be used in place of approved procedures for the determination of CATC. Under NPDWR regulations, CATC is generally measured when the total cyanide level provides a value higher than the cyanide MCL (See 57 FR 31800; July 17, 1992).

EPA–821–R–99–013, August 1999 Method OIA–1677, DW "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry,'' January 2004 is technically equivalent to Method OIA–1677, which is currently approved for determination of available cyanide in the NPDES program (64 FR 73414; December 30, 1999). Method OIA–1677, DW only differs from OIA–1677 in having (a) updated contact information, and (b) less method modification flexibility (references to performance-based modifications have been removed). Therefore the validation data on OIA–1677 is applicable to OIA–1677, DW.

Method OIA–1677 was validated by an intralaboratory validation study and a nine-laboratory validation study. The intralaboratory study was performed to establish (1) the ability of OIA–1677 to detect and quantify 11 specific metallocyanide complexes as compared to CATC and Weak Acid Dissociable (WAD) cyanide methods, (2) the ability of OIA–1677 to identify and overcome analytical interferences, and (3) compare the precision and recovery of OIA–1677 to CATC and WAD cyanide methods. These studies showed that OIA–1677 could (1) recover up to 100% of the cyanide compounds that were detected by the CATC and WAD cyanide methods, (2) overcome most analytical interferences, and (3) provide comparable or better precision and recovery than CATC and WAD cyanide methods.

The interlaboratory study was conducted to (1) confirm the performance of OIA–1677 across multiple laboratories, (2) assess interlaboratory and matrix variability, and (3) develop QC acceptance criteria. Nine laboratories participated in the study, each analyzing an identical set of six field samples (effluents) using OIA–1677. Along with these effluent analyses, laboratories performed all the required QC analyses in OIA–1677 and an MDL study. The relative standard deviation (RSD) of results across all laboratories and all samples was 12%. The mean recoveries across all effluents tested was 96%.

ASTM Method D6888–03, which also is being proposed for use in NPDES compliance monitoring in this rule, uses a similar technology to Method OIA–1677, and is described above.

While these methods generally provide dependable results, sulfide at levels below those detected with the lead acetate paper may produce false positive signals on the amperometric detection systems used in D6888–03 and OIA–1677 (see Zheng *et al.* "Evaluation and Testing of Analytical Methods for Cyanide Species in Municipal and Industrial and Contaminated Waters,'' Environ. Sci. Technol. 2003, 37, 107–115). Lead acetate paper is generally recommended means for screening for the presence of sulfide interferences in cyanide methods, but the paper will not detect sulfides below approximately 5 ppm. For this reason, analysts suspecting a sulfide interference should test their sample with a more sensitive sulfide procedure and treat the sample accordingly.

4. Radium-226 and 228 by Gamma Spectrometry

The Environmental Resources Center (ERC) at the Georgia Institute of Technology has developed a method, "The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors.'' The method simultaneously determines the concentration of both Radium-226 and Radium-228 from a single sample aliquot. This method can significantly reduce the isolation and purification steps currently required in EPA-approved sequential methods for the measurement of these radioisotopes, potentially reducing both the labor and waste disposal costs by greater than 50 percent.

A sample has its radium isotope content preconcentrated using a sulfate coprecipitation. It is then placed into a sample container appropriate for the laboratory's gamma detection system. The prepared sample is then measured in a reproducible counting geometry for a suitable amount of time so that the collected gamma spectra demonstrates the required sensitivity, defined as a Minimum Detectable Concentration (MDC), of 1 picoCurie per liter (pCi/L) for both of the regulated contaminant radioisotopes.

Method performance was characterized using a 3-laboratory study to test the method's recovery, precision, sensitivity, and ruggedness using diverse matrices found in finished drinking waters. The results of these studies demonstrate this method has the required sensitivity, and can be expected to provide results that are at least equivalent to, or have a higher degree of recovery and precision than the current EPA-approved methods for producing these measurements.

ERC's method and a copy of the method validation study report are in the docket supporting this rule. In addition, copies of ERC's method are available from The Environmental Resources Center, Georgia Institute of Technology, 620 Cherry Street, Atlanta, GA 30332–0335, USA, Phone: 404–894–3776.

B. EPA Method for Chlorine Dioxide by Colorimetry

EPA is proposing to add a new method to 40 CFR 141.74 for the measurement of chlorine dioxide residuals. EPA Method 327.0 (USEPA 2003), which has been proposed for addition to 40 CFR 141.131 in a previous rulemaking (68 FR 49548, August 18, 2003) is an enzymatic / spectrophotometric method in which a total chlorine dioxide plus chlorite concentration is determined in an unsparged sample and the chlorite concentration is determined in a sparged sample. The chlorine dioxide concentration is then calculated by subtracting the chlorite concentration from the total.

EPA proposes to approve EPA Method 327.0 as an additional method for CT determinations when chlorine dioxide is the disinfectant residual in use. It would provide water systems with additional flexibility in monitoring the application of chlorine dioxide. EPA believes that many water plant operators will prefer the new method over the currently approved methods due to its ease of use.

The pH of the samples (sparged and unsparged) and blank are adjusted to 6.0 with a citric acid/glycine buffer. The chromophore Lissamine Green B (LGB) and the enzyme horseradish peroxidase are added. The enzyme reacts with the chlorite in the sample to form chlorine dioxide which then reacts with the chromophore LGB to reduce the absorbance of the sample at 633 nm. The absorbance of the samples and blank are determined spectrophotometrically. The difference in absorbance between the samples and the blank is proportional to the chlorite and total chlorine dioxide/chlorite concentrations in the samples.

EPA Method 327.0 offers advantages over the currently approved chlorine dioxide methods in that it is not subject to positive interferences from other chlorine species and it is easier to use.

The single laboratory detection limits presented in the method are 0.04–0.16 mg/L for chlorine dioxide. The detection limits are based on the analyses of sets of seven replicates of reagent water that were fortified with low concentrations of chlorine dioxide with and without the presence of chlorite. The standard deviation of the mean concentration for each set of samples was calculated and multiplied by the student's t-value at 99% confidence and n-1 degrees of freedom (3.143 for 7 replicates) to determine the detection limit. The recovery reported in the method for laboratory fortified blanks at concentrations of 0.2–1.0 mg/L is 102–124% for chlorine dioxide with relative standard deviations between 3.6 and 16%. Replicate analyses of drinking water samples from surface and ground water sources fortified at concentrations of approximately 1 and 2 mg/L chlorite and chlorine dioxide showed average recoveries of 91–110% with relative standard deviations of 1–9%.

Method 327.0 (EPA 815–B–03–001) is available from the Office of Ground Water and Drinking Water Technical Support Center, U.S. Environmental Protection Agency, 26 W. Martin Luther King Dr., Cincinnati, OH 45268. The method also may be viewed and downloaded from *http://www.epa.gov/ogwdw/methods/sourcalt.html.*

C. New and Updated VCSB Methods

1. ASTM

This rule proposes to approve a number of updated ASTM methods in NPDWR, and NSDWR compliance monitoring. Previously approved versions of ASTM methods will remain approved. Consult Table II in Section III.A.6.b for a list of proposed methods.

Today's rule also proposes ASTM Method D 6919–03, "Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography,'' for use in drinking water compliance monitoring. Consult Section III.A.5.b for more information.

2. Standard Methods

EPA proposes to approve a number of updated Standard Methods in NPDWR, and NSDWR compliance monitoring. Copies of all the proposed methods are in the paper docket for review (they are not included in e-docket due to copyright issues). Copies of Standard Methods are available at a nominal cost from the web site *www.standardmethods.org* or from the Standard Methods Manager, American Water Works Association, 6666 West Quincy Avenue, Denver, CO 80235, 303–347–6175, *sposavec@awwa.org.* Previously approved versions of Standard Methods will remain approved.

Consult Section III.A.6.c for a discussion of EPA's proposed numbering scheme for standard methods, and Table III of that section for a list of proposed methods. While a number of methods contain no changes from previously approved version, some incorporate technical and editorial revisions to improve user-friendliness, update references, and correct errors (methods that were revised from previous versions are indicated in Table III).

D. Withdrawal of Immunoassay Method for Atrazine

A final rule was published by EPA in the **Federal Register** on October 29, 2002 (67 FR 65888), that approved Syngenta Method AG–625 for monitoring atrazine in finished drinking water. EPA proposes to withdraw this method. The proposed withdrawal is motivated by recent reports that show interferences due to chlorine and chlorine dioxide that result in false positive detection and elevated concentrations of atrazine. This has been demonstrated when measuring the concentrations of atrazine in drinking water matrices when compared to values obtained using currently approved methods. EPA seeks comments and information regarding modifications to Syngenta Method AG–625 that would eliminate or substantially mitigate the interferences described above, or regarding conditions under which the method would be suitable for use in drinking water compliance monitoring. If EPA receives such information, the Agency may, in a subsequent notice, propose to modify this method rather than withdraw approval.

V. Request for Comment on Microbiological ATP Protocol

EPA is soliciting comments on "EPA Microbiological Alternate Test Procedure (ATP) Protocol for Drinking Water, Ambient Water, and Wastewater Monitoring Methods—Guidance'' (July 2003; EPA–821–B–03–004) (Protocol). The Protocol is a guidance document for evaluating microbiological ATPs, and was referenced in the July 21, 2003, rule promulgating methods for the analysis of microbiological contaminants in ambient waters (July 21, 2003; 68 FR 43272). EPA does not plan to codify the protocol, but is interested in receiving comments that it may consider in future revisions to the protocol.

VI. Statutory and Executive Order Reviews

A. Executive Order 12866: Regulatory Planning and Review

Under Executive Order 12866 (58 FR 51735 (October 4, 1993)), the Agency must determine whether the regulatory action is "significant'' and therefore subject to OMB review and the requirements of the Executive Order. The Executive Order defines "significant regulatory action'' as one that is likely to result in a rule that may:

(1) Have an annual effect on the economy of $100 million or more, or adversely affect in a material way the economy, a sector of the economy, productivity, competition, jobs, the environment, public health or safety, or State, local, or Tribal governments or communities;

(2) Create a serious inconsistency or otherwise interfere with an action taken or planned by another agency;

(3) Materially alter the budgetary impact of entitlements, grants, user fees, or loan programs or the rights and obligations of recipients thereof; or

(4) Raise novel legal or policy issues arising out of legal mandates, the President's priorities, or the principles set forth in the Executive Order.

It has been determined that this rule is not a "significant regulatory action'' under the terms of Executive Order

12866 and is therefore not subject to E.O. 12866 review.

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.* This rule does not impose any information collection, reporting, or recordkeeping requirements. This rule merely proposes new and updated versions of testing procedures, withdraws some older testing procedures, and proposes new sample collection, preservation, and holding time requirements.

Burden means the total time, effort, or financial resources expended by persons to generate, maintain, retain, or disclose or provide information to or for a Federal agency. This includes the time needed to review instructions; develop, acquire, install, and utilize technology and systems for the purpose of collecting, validating, and verifying information, processing and maintaining information, and disclosing and providing information; adjust the existing ways to comply with any previously applicable instructions and requirements; train personnel to be able to respond to a collection of information; search data sources; complete and review the collection of information; and transmit or otherwise disclose the information.

An Agency may not conduct or sponsor, and a person is not required to respond to a collection of information unless it displays a currently valid OMB control number. The OMB control numbers for EPA's regulations in 40 CFR are listed in 40 CFR part 9.

C. Regulatory Flexibility Act

The 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.

The RFA provides default definitions for each type of small entity. It also authorizes an agency to use alternative definitions for each category of small entity, "which are appropriate to the activities of the agency'' after proposing the alternative definition(s) in the **Federal Register** and taking comment. 5 U.S.C. secs. 601(3)–(5). In addition to the above, to establish an alternative small business definition, agencies must consult with SBA's Chief Counsel for Advocacy.

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.

For purposes of assessing the impacts of this rule on small entities for methods under the Safe Drinking Water Act, EPA considered small entities to be public water systems serving 10,000 or fewer persons. This is the cut-off level specified by Congress in the 1996 Amendments to the Safe Drinking Water Act for small system flexibility provisions. In accordance with the RFA requirements, EPA proposed using this alternative definition in the **Federal Register** (63 FR 7620, February 13, 1998), requested public comment, consulted with the Small Business Administration, and expressed its intention to use the alternative definition for all future drinking water regulations in the Consumer Confidence Reports regulation (63 FR 44511, August 19, 1998). As stated in that final rule, the alternative definition would be applied to this regulation as well.

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 proposes new and updated versions of testing procedures, withdraws some older testing procedures, and proposes new sample collection, preservation, and holding time requirements. 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 have been proposed 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 have determined that a small number of small entities that are still using the CFC–113 based oil and grease methods may need to devote resources to analyst training when they switch to hexane-based methods. However, due to the decreased availability of CFC–113 in the marketplace, we anticipate that the cost differential, if any, will soon favor the use of the hexane-based methods. The phaseout of CFC–113 based methods is required to comply with the Montreal Protocol which prohibits the use of CFC–113 based methods after December 31, 2005.

Although this proposed rule will not have a significant economic impact on a substantial number of small entities, EPA nonetheless has tried to reduce the impact of this rule on small entities. Anticipating the prohibition of CFC–113 based methods, EPA promulgated hexane-based methods in May 1999. EPA has determined that most laboratories have now switched to hexane-based oil and grease methods, making the analysis costs competitive with the CFC–113 based methods. 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

Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), Public Law 104–4, establishes requirements for Federal agencies to assess the effects of their regulatory actions on State, Tribal, and local governments and the private sector. Under section 202 of the UMRA, EPA generally must prepare a written statement, including a cost-benefit analysis, for proposed and final rules with "Federal mandates'' that may result in expenditures to State, local, and Tribal governments, in the aggregate, or to the private sector, of $100 million or more in any one year. Before promulgating an EPA rule for which a written statement is needed, section 205 of the UMRA generally requires EPA to identify and consider a reasonable number of regulatory alternatives and adopt the least costly, most cost-effective or least burdensome alternative that achieves the objectives of the rule. The provisions of section 205 do not apply when they are inconsistent with applicable law. Moreover, section 205 allows EPA to adopt an alternative other than the least costly, most cost-effective or least burdensome alternative if the Administrator publishes with the final rule an explanation of why that alternative was not adopted.

Before EPA establishes any regulatory requirements that may significantly or uniquely affect small governments, including Tribal governments, it must have developed under section 203 of the UMRA a small government agency plan. The plan must provide for the notification of potentially affected small governments, enabling officials of affected small governments to have meaningful and timely input in the development of EPA regulatory proposals with significant Federal intergovernmental mandates, and informing, educating, and advising small governments on compliance with the regulatory requirements.

This rule contains no Federal mandates (under the regulatory provisions of Title II of UMRA) for State, local, or Tribal governments or the private sector. The rule imposes no enforceable duty on any State, local, or Tribal governments or the private sector. In fact, this rule should (on the whole) save money for governments and the private sector by increasing method flexibility, and allowing these entities to reduce monitoring costs by taking advantage of innovations. Thus, today's rule is not subject to the requirements of Sections 202 and 205 of the UMRA.

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 proposes new and updated versions of testing procedures, withdraws some older testing procedures, and proposes new sample collection, preservation, and holding time requirements. Thus, today's rule is not subject to the requirements of Section 203 of UMRA.

E. Executive Order 13132: Federalism

Executive Order 13132, entitled "Federalism'' (64 FR 43255, August 10, 1999), requires EPA to develop an accountable process to ensure "meaningful and timely input by State and local officials in the development of regulatory policies that have federalism implications.'' "Policies that have federalism implications'' is defined in the Executive Order to include regulations that 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.''

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. This rule merely proposes new and updated versions of testing procedures, withdraws some older testing procedures, and proposes new sample collection, preservation, and holding time requirements. 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 rule from State and local officials.

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

Executive Order 13175, entitled "Consultation and Coordination with Indian Tribal Governments'' (65 FR 67249, November 9, 2000), requires EPA to develop an accountable process to ensure "meaningful and timely input by tribal officials in the development of regulatory policies that have tribal implications.''

"Policies that have tribal implications'' is defined in the Executive Order to include regulations that have "substantial direct effects on one or more Indian tribes, on the relationship between the Federal government and the Indian tribes, or on the distribution of power and responsibilities between the Federal government and the Indian tribes.''

This proposed rule does not have tribal implications. 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, as specified in Executive Order 13175. This rule merely proposes new and updated versions of testing procedures, withdraws some older testing procedures, and proposes new sample collection, preservation, and holding time requirements. 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 Tribal governments, EPA specifically solicits comment on this proposed rule from Tribal officials.

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

Executive Order 13045: "Protection of Children from Environmental Health Risks and Safety Risks'' (62 FR 19885, April 23, 1997) applies to any rule that: (1) Is determined to be "economically significant'' as defined under Executive Order 12866, and (2) concerns an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children. If the regulatory action meets both criteria, the Agency must evaluate the environmental health or safety effects of the planned rule on children, and explain why the planned regulation is preferable to other potentially effective and reasonably feasible alternatives considered by the Agency. This proposed rule is not subject to the Executive Order 13045 because it is not economically significant as defined in Executive Order 12866. Further it does not concern an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children. This action proposes new and updated versions of testing procedures, withdraws some older testing procedures, and proposes new sample collection, preservation, and holding time requirements.

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

This rule 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

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 proposes to use over 150 standards developed by Standard Methods and ASTM International. Paragraphs III.A.3, III.A.5, III.A.6.b, and III.A.6.c specify the methods from these two voluntary consensus standards bodies (including version numbers and dates), provide information on how to obtain copies of these standards, and describe EPA's rationale for employing these standards.

List of Subjects

40 CFR Part 122

Administrative practice and procedure, Confidential business information, Environmental protection, Hazardous substances, Reporting and recordkeeping requirements, Water pollution control.

40 CFR Part 136

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

40 CFR Part 141

Chemicals, Environmental protection, Incorporation by reference, Indians-lands, Intergovernmental relations, Radiation protection, Reporting and recordkeeping requirements, Water supply.

40 CFR Part 143

Chemicals, Environmental protection, Incorporation by reference, Indians-lands, Water supply.

40 CFR Part 403

Confidential business information, Environmental protection, Reporting and recordkeeping requirements, Waste treatment and disposal, Water pollution control.

40 CFR Part 430

Environmental protection, Paper and paper products industry, Reporting and recordkeeping requirements, Waste treatment and disposal, Water pollution control.

40 CFR Part 455

Chemicals, Environmental protection, Packaging and containers, Pesticides and pests, Waste treatment and disposal, Water pollution control.

40 CFR Part 465

Coil coating industry, Environmental protection, Reporting and recordkeeping requirements, Waste treatment and disposal,Water pollution control.

Dated: March 16, 2004.

Michael O. Leavitt,

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 122—EPA ADMINISTERED PERMIT PROGRAMS: THE NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

1. The authority citation for Part 122 continues to read as follows:

Authority: The Clean Water Act, 33 U.S.C. 1251 *et seq.*

2. Section 122.1 is amended by revising paragraph (a)(4).

§ 122.1 Purpose and Scope.

(a) * * *

(4) The NPDES permit program has separate additional provisions that are used by permit issuing authorities to determine what requirements must be placed in permits if issued. These provisions are located at parts 125, 129, 133, 136 of this chapter and 40 CFR subchapter N and subchapter O of this chapter.

* * * * *

3. Section 122.21 is amended:

a. By revising paragraph (g)(7)(i).

b. In paragraph (h)(4)(i) by revising the fourth and fifth sentences with two revised sentences.

§ 122.21 Application for a permit (applicable to State programs, see § 123.25).

* * * * *

(g) * * *

(7) *Effluent Characteristics.* (i) Information on the discharge of pollutants specified in this paragraph (g)(7) (except information on storm water discharges which is to be provided as specified in § 122.26). When "quantitative data'' for a pollutant are required, the applicant must collect a sample of effluent and analyze it for the pollutant in accordance with analytical methods approved under part 136 of this chapter unless a method is specified for an industry-specific waste stream at 40 CFR subchapters N or O. When no analytical method is approved under part 136 or specified under subchapters N or O, the applicant may use any suitable method but must provide a description of the method. When an applicant has two or more outfalls with substantially identical effluents, the Director may allow the applicant to test only one outfall and report that the quantitative data also apply to the substantially identical outfall. The requirements in paragraphs (g)(7) (vi) and (vii) of this section that an applicant must provide quantitative data for certain pollutants known or believed to be present do not apply to pollutants present in a discharge solely as the result of their presence in intake water; however, an applicant must report such pollutants as present. Grab samples must be used for pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, sulfide, fecal coliform, fecal streptococcus, and volatile organics, unless specified otherwise at 40 CFR part 136. For all other pollutants, a 24-hour composite sample, using a minimum of four (4) grab samples, must be used unless specified otherwise at 40 CFR part 136. Results of analyses of individual grab samples for any parameter may be averaged to form the daily average. Grab samples that are not required to be analyzed immediately (see Table II at 40 CFR part 136) may be composited in the laboratory, provided that container, preservation, and holding time requirements are met (see Table II at 40 CFR part 136) and that sample integrity is not compromised by compositing. However, a minimum of one grab sample may be taken for effluents from holding ponds or other impoundments with a retention period greater than 24 hours. In addition, for discharges other than storm water discharges, the Director may waive composite sampling for any outfall for which the applicant demonstrates that the use of an automatic sampler is infeasible and that the minimum of four (4) grab samples will be a representative sample of the effluent being discharged.

* * * * *

(h) * * *

(4) * * *

(i) * * * Grab samples must be used for pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, sulfide, fecal coliform, fecal streptococcus, and volatile organics, unless specified otherwise at 40 CFR part 136. For all other pollutants, a 24-hour composite sample, using a minimum of four (4) grab samples, must be used unless specified otherwise at 40 CFR part 136. * * *

* * * * *

4. Section 122.41 is amended by revising paragraphs (j)(4) and (l)(4)(ii).

§ 122.41 Conditions applicable to all permits (applicable to State programs, see § 123.25).

* * * * *

(j) * * *

(4) Monitoring must be conducted according to test procedures approved under 40 CFR part 136 or unless a method is specified for an industry-

specific waste stream at 40 CFR subchapters N or O.

* * * * *

(l) * * *

(4) * * *

(ii) If the permittee monitors any pollutant more frequently than required by the permit using test procedures approved under 40 CFR part 136, or a method specified for an industry-specific waste stream at 40 CFR subchapters N or O, the results of such monitoring shall be included in the calculation and reporting of the data submitted in the DMR or sludge reporting form specified by the Director.

* * * * *

5. Section 122.44 is amended by revising paragraph (i)(1)(iv).

§ 122.44 Establishing limitations, standards, and other permit conditions (applicable to State NPDES programs; see § 123.25) .

* * * * *

(i) * * *

(1) * * *

(iv) According to test procedures approved under 40 CFR part 136 for the analyses of pollutants having approved methods under that part, unless a method is specified for an industry-specific waste stream at 40 CFR subchapters N or O; otherwise, monitoring must be conducted according to a test procedure specified in the permit for pollutants with no methods approved under 40 CFR part 136 or specified at 40 CFR subchapters N or O.

* * * * *

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 136.3 is amended:

a. In paragraph (a) by revising the introductory text and Tables IA, IB, IC, ID, and IE.

b. In paragraph (a) by adding Table IG after the notes of Table IF.

c. In paragraph (b) by revising references 6, 10, and 17, and adding references 63 through 69.

d. By revising paragraphs (c), (d), and (e).

The revisions and additions read as follows:

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, and IG. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG. 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. You can get information about obtaining these documents from the EPA Office of Water Statistics and Analytical Support Branch at 202–566–1000. Documents may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue, NW., Room B135, Washington, DC (Telephone: 202–566–2426); or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC. 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, IE, IF, and IG or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under certain circumstances (paragraph (b) or (c) of this section or 40 CFR 401.13) other test procedures may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS

Parameter and units	Method[1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods on-line	AOAC, ASTM, USGS	Other
Bacteria:						
1. Coliform (fecal) number per 100 mL.	Most Probable Number (MPN), 5 tube 3 dilution, or.	p. 132[3]	9221C E	9221C E-99		
	Membrane filter (MF)[2] single step.	p. 124[3]	9222D	9222D-97	B-0050-85[5]	
2. Coloform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 132[3]	9221C E	9221C E-99		
	MF, single step[6]	p. 124[3]	9222D	9222D-97		
3. Coliform (total), number per. 100 mL.	MPN, 5 tube, 3 dilution, or.	p. 114[3]	9221B	9221B-99		
	MF[2], single step or two step.	p. 108[3]	9222B	9222B-97	B–0025-85[5]	
4. Colofirm (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution	p. 114[3]	9221B	9221B-99		
	or MF[2] with enrichment	p. 111[3]	9222 (B+B.5 c)	9222 (B+B.5c)--97		
5. *E. coli*, number per 100 mL[28].	MPN[7, 9, 15], multiple tube		9221B.1/ 9221F[12, 14]	9221B.1/9221F–99[12, 14]		

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS—Continued

Parameter and units	Method[1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods on-line	AOAC, ASTM, USGS	Other
	multiple tube/multiple well.		9223B[13]	9223B-97[13]	991.15[11]	Coliert®[13, 17,] Colilert-18®[13 16 17]
	MF[2, 6, 7, 8, 9], two step, or.	1103.1[20]	9222B/9222G[19], 9213D	9222B/9222G-97[19]	D5392-93[10]	
	single step	1603[21], 1604[22]				mColiBlue-24[18]
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution	p. 139[3]	9230B	9230B-93		
	MF[2], or	p. 136[3]	9230C	9230C-93	B-0055-85[5]	
	Plate count	p. 143[3]				
7. Enterococci, number per 100 mL[28].	MPN[7, 9] multiple tube ...		9230B	9230B-93		
	multiple tube/multiple well.			D6503-99[10]	Enterolert®[13, 23]	
	MF[2 6 7 8 9] two step	1106.1[24]	9230C	9230C-93	5259-92[10]	
	single step, or Plate count.	1600[25], p. 143[3]				
Protozoa:						
8. *Cryptosporidium*[28].	Filtration/IMS/FA	1622[26], 1623[27]				
9. *Giardia*[28]	Filtration/IMS/FA	1623[27]				
Aquatic Toxicity:						
10. Toxicity, acute, fresh water organisms, LC50 percent effluent..	*Ceriodaphnia dubia* acute.	2002.0[29]				
	Daphnia puplex and *Daphnia magna* acute.	2021.0[29]				
	Fathead Minnow, *Pimephales promelas,* and Bannerfin shiner, *Cyprinella leedsi,* acute.	2000.0[29]				
	Rainbow Trout, *Oncorhynchus mykiss*, and brook trout, *Salvelinus fontinalis*, acute.	2019.0[29]				
	Bioluminescent bacteria, *Vibrio Fischeri*.	Microtox®[32]				
11. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC50, percent effluent..	Mysid, *Mysidopsis bahia*, acute.	2007.0[29]				
	Sheepshead Minnow, *Cyprinodon variegatus*, acute.	2004.0[29]				
	Silverside, *Menidia beryllina, Menidia menidia, and Menidia peninsulae*, actue.	2006.0[29]				
	Bioluminescent bacteria, *Vibrio Fischeri*.	Microtox®[33]				
12. Toxicity, chronic, fresh water organisms, NOEC or IC25, percent effluent..	Fathead minnow, *Pimephales promelas*, larval survival and growth.	1000.0[30]				
	Fathead minnow, *Pimephales promelas*, embryo-larval survival and teratogenicity.	1001.0[30]				

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS—Continued

Parameter and units	Method[1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods on-line	AOAC, ASTM, USGS	Other
	Daphnia, *Ceriodaphnia dubia*, survival and reproduction.	1002.0[30]				
	Green alga, *Selenastrum capricomutum*, growth.	1003.0[30]				
13. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC25, percent effluent..	Sheepshead minnow, *Cyprinodon variegatus*, larval survival and growth.	1004.0[31]				
	Sheepshead minnow, *Cyprinodon variegatus*, embryo-larval survival and teratogenicity.	1005.0[31]				
	Inland silverside, *Menidia beryllina*, larval survival and growth.	1006.0[31]				
	Mysid, *Mysidopsis bahia,* survival, growth, and fecundity.	1007.0[31]				
	Sea urchin, *Arbacia punctulata,* fertilization.	1008.0[31]				

Notes to Table IA:

[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] [Reserved]

[5] 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 Interior, Reston, VA.

[6] 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.

[7] 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.

[8] When the MF method has not be used previously to test ambient waters with high tubidity, large number 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.

[9] 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.

[10] ASTM. 2000, 1999, 1996. *Annual Book of ASTM Standards—Water and Environmental Technology.* Section 11.02. American Society for Testing and Materials. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[11] 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.

[12] 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.

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

[14] After prior enrichment in a presumptive medium for total coliform using 9221B.1, 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.

[15] 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.

[16] 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.

[17] Descriptions of the Colilert®, Colilert-18®, Quanta-Tray®, and Quanta-Tray®/2000 may be obtained from IDEXX Laboratories, Inc., One IDEXX Drive, West Brook, ME 04092.

[18] A description of the mColiBlue24® test, Total Coliforms and *E. coil,* is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

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

[20] USEPA. 2002. Method 1103.1: *Eschericia coil (E. coil)* 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–02–02.

[21] USEPA. 2002. Method 1603: *Escherichia coil (E. coil)* 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–02–023.

[22] Preparation and use of MIT 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. Microbial.* 59:3534–3544 and in USEPA. 2002. Method 1604: Total Coliforms and *Escherichia coil (E. coil)* 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.

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

[24] USEPA. 2002. 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–02–021.

[25] USEPA. 2002. 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–02–022.

[26] Method 1622 uses 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. 2001. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–01–026.

[27] Method 1623 uses 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. 2001. Method 1623. *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–01–025.

[28] Recommended for enumeration of target organism in ambient water only.

[29] 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.

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

[31] USEPA. October 2002. Methods for Measuring 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.

[32] EPA has determined that Microtox® is appropriate for use as a screening level test in a tiered testing system when used in conjunction with current EPA approved WET test methods. Microtox® may not be used by itself to monitor compliance with WET permit limits established for discharges to freshwater.

[33] Prior to using Microtox® to monitor compliance with a WET permit limit, the effluent must be tested using three different species (one test being Microtox®), and Microtox® must be determined to be the method with the most sensitive test species. This requirement strengthens the recommedation from EPA’s Technical Support Document (1991 Technical Support Document for Water Quality-based Toxics Control, Second Printing, pg. 16) which states: “To provide sufficient information for making permitting decisions, EPA recommends a minimum number of three species, representing three different phyla (e.g., a fish, an invertebrate, and a plant) be used to test an effluent for toxicity.” In addition, EPA’s NPDES regulations at 40 CFR 122.44(d)(1)(ii) require that when a permitting authority is determining WET reasonable potential for exceeding a narrative or numeric criteria within a State water quality standard, the permitting authority shall use procedures which account for (in addition to other requirements listed in the regulatory cite) the sensitivity of the species to toxicity testing (when evaluating WET).

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology[57]	Reference (method number or page)					
		EPA[52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods on-line	ASTM	USGS/AOAC/other
1. Acidity, as $CaCO_3$, mg/L	Electrometric, endpoint or phenolphthalein endpoint.		2310 B(4a)	2310 B(4a)	2310 B(4a)–01	D1067–92, 02	I–1020–85[2]
2. Alkalinity, as $CaCO_3$, mg/L	Electrometric or Colorimetric titration to pH 4.5, manual, or		2320 B	2320 B	2320 B–97	D1067–92, 02	973.43[3], I–1020–85[2]
	automatic	310.2 (Rev. 1974)[1]					I–2030–85[2]
3. Aluminum—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 D		3111 D–99		I–3051–85[2]
	AA furnace		3113 B		3113 B–99		
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4(1994)	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	Direct Current Plasma (DCP)[36].					D4190–94, 99	Note 34
	Colorimetric (Eriochrome cyanine R).		3500–Al D	3500–Al–B	3500–Al–B–01		
4. Ammonia (as N), mg/L	Manual, distillation (at pH 9.5)[6] followed by:	350.1, Rev. 2.0 (1993)	4500–NH_3 B	4500–NH_3 B	4500–NH_3 B–97		973.49[3]
	Nesslerization		4500–NH_3 C (18th only)			D1426–98, 03 (A)	973.49[3], I–3520–85[2]
	Titration		4500–NH_3 C (19th) and 4500–NH_3 E (18th)	4500–NH_3 C	4500–NH_3 C–97		
	Electrode		4500–NH_3 D or E (19th) and 4500–NH_3 F or G (18th)	4500–NH_3 D or E	4500–NH_3 D or E–97	D1426–98, 03 (B)	
	Automated phenate, or	350.1, Rev. 2.0 (1993)	4500–NH_3 G (19th) and 4500–NH_3 H (18th)	4500–NH_3 G	4500–NH_3 G–97		I–4523–85[2]
	Automated electrode						Note 7
5. Antimony—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B		3111 B–99		
	AA furnace		3113 B		3113 B–99		
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
6. Arsenic—Total,[4] mg/L	Digestion[4] followed by:	206.5 (Issued 1978)[1]					
	AA gaseous hydride		3114 B 4.d		3114 B 4.d–97	2972–97, 03 (B)	I–3062–85[2]
	AA furnace		3113 B		3113 B–99	D2972–97, 03 (C)	I–4063–98[49]
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	Colorimetric (SDDC)		3500-As C	3500-As B	3500-As B–97	2972–97, 03(A)	I–3060–85
7. Barium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 D		3111 D–99		I–3084–85[2]
	AA direct furnace[36]		3113 B		3113 B–99	D4382–95, 03	

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[57]	Reference (method number or page)					
		EPA[52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods on-line	ASTM	USGS/AOAC/other
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP[36]						Note 34
8. Beryllium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99	3645–98(88), 03(A)	I–3095–85[2]
	AA furnace		3113 B		3113 B–99	D3645–98(88), 03(B)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP, or					D4190–94, 99	Note 34
	Colorimetric (aluminon)		3500–Be D				
9. Biochemical oxygen demand (BOD$_5$), mg/L:	Dissolved Oxygen Depletion		5210 B	5210 B	5210 B–01		973.44,[3] p. 17.[9], I–1578–78[8], I–3112–85[2], I–4471–97[50]
10. Boron[37]—Total, mg/L:	Colorimetric (curcumin)		4500–B	4500–B	4500–B B–00		
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	DCP					D4190–94, 99	Note 34
11. Bromide, mg/L:	Titrimetric					D1246–95(99)(C)	p. S44.[10], I–1125–85[2]
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						Waters[54]
12. Cadmium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D3556–97, 02 (A or B)	974.27,[3] p. 37.[9], I–3135–85[2] or I–3136–85[2]
	AA furnace		3113 B		3113 B–99	D3557–95, 02 (D)	I–4138–89[51]
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–1472–85[2] or I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP[36]					D4190–94, 99	Note 34
	Voltametry[11], or					D3557–95, 02 (C)	
	Colorimetric (Dithizone)		3500–Cd D		3500–Cd D–90		
13. Calcium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99	D511–93, 03(B)	I–3152–85[2]
	ICPA/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–4471–97[50]
	DCP, or						Note 34
	Titrimetric (EDTA)		3500–Ca D	3500–Ca B	3500–Ca B–97	D511–93, 03(A)	
	Ion Chromatography					D 6919–03	
14. Carbonaceous Biochemical oxygen demand (CBOD$_5$), mg/L$_{12}$.	Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B	5210 B	5210 B–01		
15. Chemical oxygen demand (COD), mg/L	Titrimetric	410.3 (Rev. 1978)[1]	5220 C	5220 C	5220 C–97	D1252–95, 00 (A)	973.46,[3] p. 17.[9] I–3560–85[2]

	Spectrophotometric, manual or automatic.	410.4, Rev. 2.0 (1993)	5220 D	5220 D	5220 D–97	D1252–95, 00 (B)	Notes 13, 14. I–3561–85[2]
16. Chloride, mg/L	Titrimetric (silver nitrate) or		4500–Cl B	4500–Cl B	4500–CL B–97	D512–89 (99) (B)	I–1183–85[2]
	(Mercuric nitrate)		4500–Cl–C	4500–Cl–C	4500–Cl–C–97	D512–89(99) (A)	973.51,[3] I–1184–85[2]
	Colorimetric, manual or						I–1187–85[2]
	Automated (Ferricyanide)		4500–Cl–E	4500–Cl–E	4500–Cl–E–97		I–2187–85[2]
	Potentiometric Titration		4500–Cl–D	4500–Cl–D	4500–Cl–D–97		
	Ion Selective Electrode					D512–89(99) (C)	
	Ion Chromatography	300.0 Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97	993.30[3]
	CIE/UV						Waters[54]
17. Chlorine—Total residual, mg/L; Titrimetric:	Amperometric direct, or		4500–Cl D	4500–Cl D	4500–Cl D–00		
	Amperometric direct (low level)		4500–Cl E	4500–Cl E	4500–Cl E–00		
	Iodometric direct		4500–Cl B	4500–Cl B	4500–Cl B–00		
	Back titration ether end-point[15] or.		4500–Cl C	4500–Cl C	4500–Cl C–00		
	DDP–FAS		4500–Cl F	4500–Cl F	4500–Cl F–00		
	Spectriogitinetrucm		4500–Cl G	4500–Cl G	4500–Cl G–00		
	DPD						
	Or Electrode						Note 16
18. Chromium VI dissolved, mg/L	0.45 micron Filtration followed by:						
	AA chelatoin-extraction or		3111 C		3111 C–99		I–1232–85
	Ion Chromatography	218.6, Rev. 3.3 (1994)	3500–Cr E	3500–Cr C	3500–Cr C–01	D5257–97	993.23
	Colorimetric (Diphenyl-carbazide).		3500–Cr D	3500–Cr B	3500–CR B–01	D1687–92, 02 (A)	I–1230–85
19. Chromium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspriation[36]		3111B		3111 B–99	D1687–92, 02 (B)	974.27[3], I–3236–95[2]
	AA chelation-extraction		3111 C		3111 C–99		
	AA furnace		3113 B		3113 B–99	D1687 92, 02 (C)	I–3233–93[46]
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev/ 4/4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP,[36]					D4190–94, 99	Note 34
	Colorimetric (Diphenylcarbazide).		3500–Cr D	3500–Cr B	3500–Cr B 01		
20. Cobalt—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspriation		3111 B or C		3111 B or C–99	D3558–94, 03 (A or B)	p. 37[9], I–3239–85[2]
	AA furnace		3113 B		311 B–99	D3558–94, 03 (C)	I–4243–89[51]
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP					D4190–94, 99	Note 34
21. Color platinum cobalt units or dominant wave-length, hue luminance purity:.	Colorimetric (ADMI), or		2120 E	2120E			Note 18
	(Platinum cobalt), or		2120 B	2120 B	2120 B–01		I–1250–85[2]
	Spectrophotometric		2120 C	2120 C			
Copper-Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D1688–95, 02 (A or B)	974.27[3] p. 37.[9] I–3270–85[2] or I–3271–85[2]
	AA furnace		3113 B		3113 B–99		I–4274–89[51]
	STGFAA	200.9, Rev. 2.2 (1994)					

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[57]	Reference (method number or page)					
		EPA[52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods on-line	ASTM	USGS/AOAC/other
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B		3120 B–99		I4471–97[50]
	ICP/MS	200.8 Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP[36] or				D4190–94, 99	Note 34	
	Colorimetric (Neocuproine) or		3500–Cu D	3500–Cu B	3500–Cu B–99		
	Bicinchoninate)		3500–Cu B	3500–Cu B	3500–Cu C–99		Note 19
23. Cyanide-Total, mg/L:	Automated Distillation and Colorimetry, or.						Kelada–01[55]
	Manual distillation with $MgCl_2$ followed by.	335.4 Rev. 1.0 (1993)[57]	4500–CN–C	4500–CN–C		D2036–98(A)	10–204–00–1–X[56]
	Titrimetric, or		4500–CN–D	4500–CN–D	4500–CN–D–99		p. 22[9]
	Spectrophotometric, manual or		4500–CN–E	4500–CN–E	4500–CN–E–99	D2036–98(A)	I–3300–85
	Automated[20], or	335.4, Rev. 1.0 (1993)[57]					10–204–00–1–X[56], 1–4302–85[2]
	Ion Selective Electrode		4500–CN–F	4500–CN–F	4500–CN–F–99	D2036–98(A)	
24. Available Cyanide, mg/L:	Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$ followed by Titrimetric or Spectrophotometric.		4500–CN–G	4500–CN–G	4500–CN–G–99	D2036–98(B)	
	Flow injection and ligand exchange, followed by amperometry..					D6888–03	OIA–1677[44]
	Automated Distillation and Colorimetry.						Keleda–01[55]
25. Fluoride—Total, mg/L:	Manual distillation[6] followed by		4500–F–B	4500–F–B	4500–F–B–97		
	Electrode, manual or				4500F–C–97	D1179–93, 99 (B)	
	Automated or						I–4327–85[2]
	Colorimetric (SPADNS) or		4500–F–D	4500–F–D	4500–F–D–97	D1179–93, 99 (A)	
	Automated complexone		4500–F–E	4500–F–E	4500–F–E–97		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1 Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97,03	993.30[3]
	CIE/UV						Waters[54]
26. Gold—Total[4] mg/L	Digestion[4] followed by:.						
	AA direct aspiration, or		3111 B		3111 B–99		
	AA furnace, or	231.2 (Rev. 1878)[1]					
	STGFAA	200.9 Rev. 2.2 (1994)					
	DCP						Note 34
27. Hardness—Total, as $CaCO_3$, mg/L	Automated colorimetric,	130.1 (Issued 1971)[1]					
	Titrimetric (EDTA), or		2340 B or C	2340 B or C	2340 B or C–97	D1136–86(92), 02	973.52B.[3], I–1338–85[2]
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).						
28. Hydrogen ion (pH), pH units	Electrometric measurement, or		4500–H^+ B	4500–H^+ B	4500–H^+ B–00	D1293–84 (90), 99 (A or B)	973.41.[3], I–1586–85[2]
	Automated electrode	150.2 (Dec. 1982)[1]					Note [21, I–2587–85[2]
29. Iridium—Total,[4] mg/L	Digestion[4] followed by:.						
	AA direct aspiration or		3111 B		3111 B–99		
	AA furnace	235.2 (Issued 1978)[1]					
30. Iron—Total,[4] mg/L	Digestion[4] followed by:.						

	AA direct aspiration[36]		3111 B or C		3113 B–89, 93, 99	D1068–96, 03 (A or B)	974.27.[3], I–3381–85[2]
	AA furnace		3113 B		3113 B–99	D 1068–96, 03 (C)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–4471–97[50]
	DCP[36]					D4190–94, 99	Note 34
	Colorimetric (Phenanthroline)		3500–Fe D	3500–Fe B	3500–Fe B–97	D1068–96, 03 (D)	Note 22
31. Kjeldahl Nitrogen[5]—Total, (as N), mg/L	Digestion and distillation followed by:[20].		4500–N_{org} B or C with 4500–NH_3 B	4500–N_{org} B or C with 4500–NH_3 B	4500–N_{org} B or C–97 with 4500–NH_3 B–97	D3590–89, 02 (A)	
	Titration, or		4500–NH_3 C (19th) and 4500–NH_3 E (18th)	4500–NH_3 C	4500–NH_3 C–97	D3590–89, 02 (A)	973.48[3]
	Nesslerization, or		4500–NH_3 C (18th Only)			D3590–89, 02 (A)	
	Electrode		4500–NH_3 F or G (18th) and 4500–NH_3 D or E (19th)	4500–NH_3 D or E	4500–NH_3 D or E–97		
	Automated phenate colorimetric.	351.1 (Rev. 1978)[1]					I–4551–78[8]
	Semi-automated block digestor colorimetric.	351.2, Rev. 2.0 (1993)				D3590–89, 02(B)	I–4515–91[45]
	Manual or block digestor potentiometric.					D3590–89, 02 (A)	
	Block digester, followed by Auto distillation and Titration, or.						Note 39
	Nesslerization, or						Note 40
	Flow injection gas diffusion						Note 41
32. Lead—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D3559–96, 03 (A or B)	[3]974.27, [2]I–3399–85
	AA furnace		3113 B		3113 B–99	D3559–96, 03 (D)	[51]I–4403–89
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		[50]I–4471–97
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	[3]993.14
	DCP					D4190–94, 99	Note 34
	Voltametry[11] or					D3559–96, 03 (c)	
	Colorimetric (Dithizone)		3500–Pb D	3500–Pb B	3500–Pb B–97		
33. Magnesium—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99	D511–93, 93 (b)	[3]974.27, [2]I–3447–85
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		[50]I–4471–97
	DCP or						Note 34
	Gravimetric		3500–Mg D				
	Ion Chromatography					D6919–03	
34. Manganese—Total,[4] mg/L	Digestion[4] followed by:						
	AA direct Aspiration[36]		3111 B		3111 B–99	D858–95 (A or B)	[3]974.27, [2]I–3454–85
	AA furnace		3113 B		3113 B–99	D858–95, 02 (c)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		[50]I–4471–97
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	[3]993.14
	DCP[36], or					D4190–94, 99	Note 34
	Colorimetric (Persulfate), or		3500–Mn D	3500–Mn B	3500–Mn B–99		[3]920.203
	(Periodate)						Note 23

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[57]	Reference (method number or page)					
		EPA[52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods on-line	ASTM	USGS/AOAC/other
35. Mercury—Total[4], mg/L:	Cold vapor, manual or	245.1, Rev. 3.0 (1994)	3112 B		3112 B–99	D3223–91	[3]977.22, [2]I3462–85
	Automated	245.2 (Issued 1974)					
	Cold vapor atomic fluorescence spectrometry (CVAFS).	245.7 (Dec. 2003)[58]					
	Purge and Trap CVAFS	1631E[43]					
Molybedenum—Total[4], mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99		[2]I–3490–85
	AA furnace		3113 B		3113 B–99		[47]I–3492–96
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B		3120 B–99		[50]I–4471–97
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	[3]993.14
	DCP						Note 34
37. Nickel-Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D1886–90, 94 (98) (A or B)	I–3499–85[2]
	AA furnace		3113 B		3113 B–99	D1886–90, 94 (98)(C)	I–4503–89[51]
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)					
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP[36], or					D4190–94, 99	Note 34
	Colorimetric (heptoxime)		3500–NI D (17th Edition)				
38. Nitrate (as N), mg/L	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						Waters[54]
	Ion Selective Electrode		4500 NO_3 minus; D	4500 NO_3 minus; D	4500 NO_3 minus; D–00		
	Colorimetric (Brucine sulfate), or	352.1[1]					97.50[3] 419D[17], p. 28.[9]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).						
39. Nitrate-nitrite (as N), mg/L	Cadmimum reduction, Manual or		4500–NO_3 minus; E	4500–NO_3 minus; E	4500–NO_3 minus; E–00	D3867–99(B)	
	Automated, or	353.2, Rev. 2.0 (1993)	4500–NO_3 minus; F	4500–NO_3 minus; F	4500–NO_3 minus; F–00	D3867–99(A)	I–4545–85[2]
	Automated hydrazine		4500–NO_3 minus; H	4500–NO_3 minus; H	4500–NO_3 minus; H–00	D4327–97	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	4327–97, 03	993.30[3]
	CIE/UV						Waters[54]
40. Nitrite (as N), mg/L	Spectrophoto-metric: Manual or		4500–NO_2 B	4500–NO_2 B	4500–NO_2 B–00		Note 25
	Automated (Diazotization)						I–4540–85[2]
	Automated (*bypass cadmium reduction).	353.2, Rev. 2.0 (1993)	4500–NO_3 minus; F	4500–NO_3 minus; F	4500–NO_3 minus; F–00	D3867–99(A)	
	Manual (* bypass cadmium reduction).		4500–NO_3 minus; E	4500–NO_3 minus; E	4500–NO_3 minus; E–00		

	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]
	CIE/UV						Waters[54]
41. Oil and grease—Total recoverable, mg/L	Hexane extractable material (HEM); n-Hexane extraction and gravimetry.	1664A[42]	5520 B[38]	5520 B[38]	5520 B–01[38]		
	Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry.	1664A[42]					
42. Organic carbon—Total (TOC), mg/L	Combustion or oxidation		5310 B, C, or D	5310 B, C, or D	5310 B, C, or D–00	2579–93 (A or B)	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, or	365.1, Rev. 2.0 (1993)	4500–P F	4500–P F			973.56[3], 1–4601–85[2]
	Manual single reagent		4500–P E	4500–P E		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	4110 B	4110 B–00	D4327–97, 03	993.30[33]
	CIE/UV						Waters[54]
45. Osmium-Total[4], mg/L	Digestion[4] followed by:						
	AA direct aspiration, or		3111 D		3111 D–99		
	AA furnace	252.2 (Issued 1978)[1]					
46. Oxygen, dissolved, mg/L	Winkler (Azide modification), or		4500–O C	4500–O C	4500–O C–01	888–92, 03 (A)	I–1576–78[8]
	Electrode		4500–O G	4500–O G	4500–O G–01	D888–92, 03 (B)	1–1576–78[8]
47. Palladium—Total,[4] mg/L	Digestion[4] followed by						
	AA direct aspiration, or		3141 B		3111 B–99		p. S27.[10]
	AA furnace	253.2[1] (Issued 1978)					p. S28.[10]
	DCP						Note 34.
48. Phenois, mg/L	Manual distillation[26] Followed by:.	420.1[1] (Rev. 1978)					Note 27.
	Colorimetric (4AAP) manual, or	420.1[1] (Rev. 1978)					Note 27.
	Automated	420.4 Rev. 1.0 (1993)					
49. Phosphorus (elemental), mg/L	Gas-liquid chromatography						Note 28.
50. Phosphorus—Total, mg/L	Persulfate digestion followed by:[29].		4500–P B.5	4500–P B.5			973.55[3]
	Manual or	365.3[1] (Issued 1978)	4500–P E	4500–P E		D515–88(A)	
	Automated ascorbic acid reduction.	365.1 Rev. 2.0 (1993)	4500–P F	4500–P F			973.56[3], I–400–85[2]
	Semi-automated block digestor	365.4[1] (Issued 1974)				D515–88(B)	I–4610–91[48]
51. Platinum-Total,[4] mg/L	Digestion[4] followed by:.						
	AA direct aspiration		3111 B		3111 B–99		
	AA furnace	255.2[1]					
	DCP						Note 34
52. Potassium-Total,[4] mg/L:	Digestion[4] followed by:.						
	AA direct aspiration		3111 B		3111 B–99		973.l53[3], 1–3630–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	Flame photometric or		3500–K D	3500–K B	3500–K B–99		
	Colorimetric						317 B[17]
	Ion Chromatography					D 6919–03	
53. Residue-Total, mg/L	Gravimetric, 103–105°		2540 B	2540 B	2540 B–97		I–3750–85[2]
54. Residue-filterable, mg/L	Gravimetric, 180°		2540 C	2540 C	2540 C–97		I–1750–85[2]

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology[57]	Reference (method number or page)					
		EPA[52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods on-line	ASTM	USGS/AOAC/other
55. Residue-non-filterable (TSS), mg/L	Gravimetric, 103–105° post washing of residue.		2540 D	2540 D	2540 D–97		I–3765–85[2]
56. Residue-settleable, mg/L:	Volumetric, (Imhoff cone), or gravimetric.		2540 F	2540 F	2540 F–97		
57. Residue-Volatile, mg/L	Gravimetric, 550°	160.4[1]					I–3753–85[2]
58. Rhodium-total,[4] mg/L	Digestion[4] followed by:.						
	AA direct aspiration, or		3111 B	3111 B	3111 B–99		
	AA furnace	265.2[1]					
	STGFAA	200.9, Rev. 2.2 (1994)					
59. Ruthenium-Total,[4] mg/L	Digestion[4] followed by:.						
	AA direct aspiration, or		3111 B		3111 B–99		
	AA furnace	267.2[1]					
	STGFAA	200.9, Rev. 2.2 (1994)					
60. Selenium-Total,[4] mg/L	Digestion[4] followed by:.						
	AA furnace		3113 B		3113 B–99	D3859–98, 03 (B)	I–4668–98[49]
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES,[36]	200.7[5], Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	AA gaseous hydride		3114 B	3114 B	3114 B–97	D3859–98, 03 (A)	I–3667–85[2]
61. Silica[37]-Dissolved, mg/L	0.45 micron filtration followed by:.						
	Colorimetric, Manual or		4500-Si D	4500-SiO_2	4500-SiO_2 C–97	D859–94, 00	I–1700–85[2]
	Automated (Molybdosilicate), or.						I–2700–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–4471–97[50]
62. Silver-Total,[4] mg/L	Digestion[4, 29] followed by:.						
	AA direct aspiration		3111 B or C		3111 B or C–99		974.27[3], p. 37[9], I–3720–85[2]
	AA furnace		3113 B		3113 B–99		I–4724–89[51]
	STGFAA	200.9, Rev. 2.2 (1994)					
		ICP/AES	200.7, Rev. 4.4 (1994)	3210 B	3120 B	3120 B–99	I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP						Note 34
63. Sodium-Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99		973.54[3], I–3735–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–4471–97[50]
	DCP, or						Note 34
	Flame photometric		3500-Na D	3500-Na B	3500-Na B–97		
	Ion Chromatography					D 6919–03	
64. Specific conductance, micromhos/cm at 25°, C.	Wheatstone bridge	120.1[1] (Rev. 1982)	2510 B	2510 B	2510 B–97	D1125–95 (99)(A)	973.40[3], I–2781–85[2]
65. Sulfate (as SO_4), mg/L	Automated colorimetric	375.2, Rev. 2.0 (1993)					
	Gravimetric		4500-SO$^{4\ -2}$ C	4500-SO$^{4\ -2}$ C			925.54.[3]
	Turbidimetric					D516–90, 02	426C.[30]

	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[33]
	CIE/UV						Waters[54]
66. Sulfide (as S), mg/L	Titrimetric (iodine), or		4500-S $^{-2}$F (19th) 4500-S $^{-2}$E (18th)	4500-S $^{-2}$F–00		I–3840–85[2]	
	Colorimetric (methylene blue)		4500-S $^{-2}$D	4500-S $^{-2}$D–00	4500-S $^{-2}$		
	Ion Selective Electrode		4500-S $^{-2}$G	4500-S $^{-2}$G	4500-S $^{-2}$G–00	4652–03	
67. Sulfite (as SO_3), mg/L	Titrimetric (iodine-iodate)		4500-SO$_3^{-2}$B	4500-SO$_3^{-2}$B	4500-SO$_3^{-2}$B–00		
68. Surfactants, mg/L	Colorimetric (methylene blue)		5540 C	5540 C	5540 C–00	D2330–88, 02	
69. Temperature, °C		Thermometric	2550 B	2550 B	2550 B–00		Note 32
70. Thallium-Total,[4] mg/L	Digestion[4] by:						
	AA direct aspiration		3111 B		3111 B–99		
	AA furnace	279.2[1] (Issued 1978)					
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02.	993.14[3]
71. Tin-Total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 B		3111 B–99		I–3850–78[8]
	AA furnace, or		3113 B		3113 B–99		
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)					
72. Titanium-total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99		
	AA furnace	283.2[1] (Issued 1978)					
	DCP						Note 34
73. Turbidity, NTU[53]	Nephelometric	180.1, Rev. 2.0 (1993)	2130 B	2130 B	2130 B–01	D1889–94, 00 (A)	I–3860–85[2]
74. Vanadium-total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration		3111 D		3111 D–99		
	AA furnace					D3373–93, 03	
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99		I–4471–97[50].
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673–02	993.14[3]
	DCP, or					D4190–94, 99	Note 34.
	Colorimetric (Gallic Acid)		3500–V D	3500–V B	3500–V B–97		
75. Zinc-total,[4] mg/L	Digestion[4] followed by:						
	AA direct aspiration[36]		3111 B or C		3111 B or C–99	D1691–95, 02 (A or B)	974.27[3], p. 37[9], I–3900–85[2].
	AA furnace	289.2[1] (Issued 1978)					
	ICP/AES[36]	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B–99[59]		I–4471–97[50].
	ICP/MS	200.8, Rev. 5.4 (1994)			D5673–02	993.14[3]	
	DCP,[36] or					D4190–94, 99	Note 34
	Colorimetric (Dithizone) or		3500–ZN E				
	(Zincon)		3500–Zn F	3500–Zn B	3500–ZN B–97		Note 33.

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, 16th ed.

[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 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 approval 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 must 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.

[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, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

[10] 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).

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

[12] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test method with measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data using a nitrification inhibitor.

[13] OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

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

[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 approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0± 0.2. The approved methods are given on pp 576–81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrometric procedure.

[28] R.F. Addition 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 approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition.

[31] EPA Methods 335.2 and 335.3 require the NaOH absorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.

[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] Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals".

[36] "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 extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Method 1664A). 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, PO 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, PO Box 9010, College Station, TX 77842.

[41] Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, PO Box 9010, College Station, TX 77842.

[42] Method 1664, Revision A "n–Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n–Hexane Extractable Material (SGT–HEM; Non-polar Material) by Extraction and Gravimetry" EPA–821–R–98–002, February 1999. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.

[43] USEPA. 2001. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Automic 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 (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 Methods," 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] All 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;11, 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/met300.pdf*.

[53] AMCO–AEPA-1styrene divinyl beads and Hach StablCal 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, 01y757, 508/482–2131 (Office), 508/482–3625 (FAX).

[55] Kelada-01, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821–B–91–009 National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. Phone: 800–553–6847.

[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 Rd., Milwaukee, WI 53218, USA. Phone: 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] Method 245.7, "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry" [December 2003] (EPA–821–D–03–001).

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter[1]	EPA method number,[2,7]			Other Approved Methods			
	GC	GC/MS	HPLC	Standard methods [edition(s)]	Standard methods on-line	ASTM	Other
1. Acenaphthene	610	625, 1625B	610	6440 B [18th, 19th, 20th].		D4657–92(99)	Note 9, p. 27.
2. Acenaphthylene	610	625, 1625B	610	6440 B, 6410 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
3. Acrolein	603	[4]624, 1624B.					
4. Acrylonitrile	603	[4]624, 1624B.					
5. Anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
6. Benzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97.		
7. Benzidine		[5]625, 1625B	605				Note 3, p. 1.
8. Benzo(a)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
9. Benzo(a)pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
10. Benzo(b)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
11. Benzo(g,h,i)perylene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
12. Benzo(k)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
13. Benzyl chloride							Note 3, p. 130: Note 6, p. S102.
14. Benzyl butyl phthalate	606	625, 1625 B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
15. Bis(2-chloroethoxy) methane.	611	625, 1625 B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
16. Bis(2-chloroethoxy) ether.	611	625, 1625 B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
17. Bis(2-ethylhexyl) phthalate.	606	625, 1625 B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
18. Bromodichloro-methane).	601	624, 1624 B		6200 C [20th, and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97		
19. Bromoform	601	624, 1624 B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97		
20. Bromomethane	601	624, 1624 B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97		
21. 4-Bromophenylphenyl ether.	611	625, 1625 B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
22. Carbon tetrachloride	601	624, 1624 B		6200 C [20th] and 6230 B [18th, 19th].			Note 3, p. 130.
23. 4-Chloro-3-methyl-phenol.	604	625, 1625 B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		Note 9, p. 27.
24. Chlorobenzene	601, 602	624, 1624 B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		Note 3, p. 130.
25. Chloroethane	601	624, 1624B		6200 B [20TH] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		
26. 2-Chloroethylvinylether	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number,[2] [7]			Other Approved Methods			
	GC	GC/MS	HPLC	Standard methods [edition(s)]	Standard methods on-line	ASTM	Other
27. Chloroform	601	624, 1624B		6200 B[20th] and 6210 B [18th, 19th]. 6200 C [20th] and 6230 B.	6200 B and C–97	....	Note 3, p. 130.
28. Chloromethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97	....	
29. 2-Chloronaphthalene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00	....	Note 9, p. 27.
30. 2-Chlorophenol	604	625, 1625B	....	6410 B, 6420 B [18th, 19th, 20th].	6410–B–00, 6420 B–00	....	Note 9, p. 27.
31. 4-Chlorophenylphenylether.	611	625, 1625B	6410 B [18th, 19th, 20th]	6410 B–00	....	Note 9, p. 27..	
32. Chrysene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B–00	4657–92(99)	Note 9, p. 27.
33. Dibenzo(a,h)anthracene.	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B–00	D4657–92(99)	Note 9, p. 27.
34. Dibromochloro-methane.	601	624, 1624B	....	6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97	....	
35. 1,2-Dichlorobenzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97	....	Note 9, p27
36. 1,3-Dichlorobenzene	601, 602	624, 1625B	....	6200C [20th] and 6220B (18th, 19th], 6200C [20th] and 6230B [18th, 19th].	6200B and C–97	....	Note 9, p. 27
37. 1,4-Dichlorobenzene	601, 602	624, 1625B	....	6200C [20th] and 6220B [18th, 19th], 6200C [20th] and 6230B [18th, 19th].	6200B and C–97	....	Note 9, p. 27
38. 3,3-Dichlorobenzidine	....	625, 1625B	605	6410B [18th, 19th, 20th].	6410B–00	....	
39. Dichlorodifluoro-methane.	601	....	....	6200C [20th] and 6230B [18th, 19th].	6200 C–97	....	
40. 1,1-Dichloroethane	601	624, 1624B	....	6200B [20th] and 6210B [18th, 19th], 6200C [20th] and 6230B [18th, 19th].	6200B and C–97	....	
41. 1,2-Dichloroethane	601	624, 1624B	....	6200B [20th] and 6210B [18th, 19th], 6200C [20th] and 6230B [18th, 19th].	6200B and C–97	....	
42. 1,1-Dichloroethene	601	624, 1624B	....	6200B [20th] and 6210B [18th, 19th], 6200C [20th] and 6230B [18th, 19th].	6200B and C–97	....	
43. trans-1,2-Dichloroethene.	601	624, 1624B	....	6200B [20th] and 6210B [18th, 19th], 6200C [20th] and 6230B [18th, 19th].	6200B and C–97	....	
44. 2,4-Dichlorophenol	604	625, 1625B	....	6410B, 6420B [18th, 19th, 20th].	6410B–00, 6420B–00.	....	Note 9, p. 27
45. 1,2-Dichloropropane	601	624, 1624B	....	6200 B [20th] and 6010 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97	....	

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number,[2 7]			Other Approved Methods			
	GC	GC/MS	HPLC	Standard methods [edition(s)]	Standard methods on-line	ASTM	Other
46. cis-1,3-Dichloropropene.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		
47. trans-1,3-Dichloropropene.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		
48. Diethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27
49. 2,4-Dimethylphenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		Note 9, p. 27
50. Dimethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27
51. Di-n-butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27
52. Di-n-octyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27
53. 2,3-Dinitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		
54. 2,4-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27
55. 2,6-Dinitrotoluene	609	625, 1625B		6410 B [16th, 19th, 20th].	6410 B–00		Note 9, p. 27
56. Epichlorohydrin							Note 3, p. 130; Note 6, p. S102.
57. Ethylbenzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97		
58. Fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657—92(99)	Note 9, p. 27
59. Fluorene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657—92(99)	Note 9, p. 27
60. 1,2,3,4,5,6,7–Heptachlorodi–benzofuran.		1613B [10]					
61. 1,2,3,4,7,8,9–Heptachlorodi–benzofuran.		1613B [10]					
62. 1,2,3,4,6,7,8–Heptachlorodibenzo–*p*–dioxin.		1613B [10]					
63. Hexachlorobenzene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
64. Hexachlorobutadiene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
65. Hexachlorocyclopentadiene.	612	625,[5] 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
66. 1,2,3,4,7,8–Hexachlorodibenzofuran.		1613B [10]					
67. 1,2,3,6,7,8–Hexachlorodibenzofuran.		1613B [10]					
68. 1,2,3,7,8,9–Hexachlorodibenzofuran.		1613B [10]					
69. 2,3,4,6,7,8–Hexachlorodibenzofuran.		1613B [10]					
70. 1,2,3,4,7,8–Hexachlorodibenzo–*p*–dioxin.		1613B [10]					
71. 1,2,3,6,7,8–Hexachlorodibenzo–*p*–dioxin.		1613B [10]					
72. 1,2,3,7,8,9–Hexachlorodibenzo–*p*–dioxin.		1613B [10]					
73. Hexachloroethane	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
74. Ideno(1,2,3–cd) pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
75. Isophorone	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter[1]	EPA method number,[2,7]			Other Approved Methods			
	GC	GC/MS	HPLC	Standard methods [edition(s)]	Standard methods on-line	ASTM	Other
76. Methylene chloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th].	6200 C–97		Note 3, p. 130
77. 2-Methyl-4,6-dinitrophenol.	604	625, 1625B		6420 B, 6410 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		Note 9, p. 27.
78. Naphthalene	610	625, 1625B	610	6440 B, 6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
79. Nitrobenzene	609	625, 1625B		6410 B [18th, 19th, 20th].	6410B–00	D4657–92(99)	Note 9, p. 27.
80. 2-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	64310 B–00, 64320 B–00.		Note 9, p. 27.
81. 4-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		Note 9, p. 27.
82. N-Nitrosodimethylamine.	607	625,[5] 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
83. N-Nitrosodi-n-propylamine.	607	625,[5] 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
84. N-Nitrosodiphenylamine.	607	625,[5] 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 9, p. 27.
85. Octachlorodibenzofuran.		1613B [10].					
86. Octachlorodibenzo-*p*-dioxin.		[10]1613B.					
87. 2,2′-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether].	611	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00.		
88. PCB-1016	608	625		6410 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 43.
89. PCB-1221	608	625		6410 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 43.
90. PCB-1232	608	625		6410 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 43.
91. PCB-1242	608	625		6410 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 43.
92. PCB-1248	608	625.					
93. PCB-1254	608	625		6410 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 43.
94. PCB-1260	608	625		6410 B, 6630 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 43.
95. 1,2,3,7,8-Pentachlorodibenzofuran.		1613B [10].					
96. 2,3,4,7,8-Pentachlorodibenzofuran.		1613B [10].					
97. 1,2,3,7,8,-Pentachlorodibenzo-*p*-dioxin.		1613B [10].					
98. Pentachlorophenol	604	625, 1625B		6410 B, 6630 B [18th, 19th, 20].	6410 B–00		Note 3, p. 140; Note 9, p. 27.
99. Phenanthrene	610	625, 1625B	610	6410 B 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	Note 9, p. 27.
100. Phenol	604	625, 1625B		6420 B, 6410 B [18th, 19th, 20th].	6410 B–00 6420 B–00		Note 9, p. 27.
101. Pyrene	610	625, 1625B	610	6440 B, 6410 B [18th, 19th, 20th].	6410 B–00	D4657–92(99)	Note 9, p. 27.
102. 2,3,7,8-Tetrachlorodibenzofuran.		1613B [10].					
103. 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin.		613, 625,[5a] 1613B [10].					
104. 1,1,2,2-Tetrachloroethane.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		Note 3, p. 130
105. Tetrachloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		Note 3, p. 130
106. Toluene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97.		
107. 1,2,4-Trichlorobenzene.	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		Note 3, p. 130; Note 9, p.27

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter[1]	EPA method number,[2] [7]			Other Approved Methods			
	GC	GC/MS	HPLC	Standard methods [edition(s)]	Standard methods on-line	ASTM	Other
108. 1,1,1-Trichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
109. 1,1,2-Trichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		Note 3, p. 130
110. Trichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
111. Trichlorofluoromethane.	601	624		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		
112. 2,4,6-Trichlorophenol	604	625, 1625 B		6420 B, 6410 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		Note 9, p. 27
113. Vinyl chloride	601	624, 1624 B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200B and C–97		

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.

[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 extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624B.

[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 TM 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. PowerPrep system in place of manual cleanup provided that analysts meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

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

Parameter	Method	EPA[2,7]	Standard methods 18th, 19th, 20th Ed.	Standard methods on-line	ASTM	Other
1. Aldrin	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
2. Ametryn	GC					Note 3, p. 83; Note 6, p. S68.
3. Aminocarb	TLC					Note 3, p. 94; Note 6, p. S16.
4. Atraton	GC					Note 3, p. 83; Note 6, p. S68.
5. Atrazine	GC					Note 3, p. 83; Note 6, p. S68; Note 9.
6. Azinphos methyl	GC					Note 3, p. 25; Note 6, p. S51.
7. Barban	TLC					Note 3, p. 104; Note 6, p. S64.
8. α-BHC	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 8.
	GC/MS	[5] 625	6410 B			
9. β-BHC	GC	608	6630 C		D3086–90, D5812–96(02)	Note 8.
	GC/MS	[5] 625	6410 B	6410 B–00		
10. δ-BHC	GC	608	6630 C		D3086–90, D5812–96(02)	Note 8.
	GC/MS	[5] 625	6410 B	6410 B–00		
11. γ-BHC (Lindane)	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		

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

Parameter	Method	EPA [2,7]	Standard methods 18th, 19th, 20th Ed.	Standard methods on-line	ASTM	Other
12. Captan	GC		6630 B		D3086–90, D5812–96(02)	Note 3, p. 7.
13. Carbaryl	TLC					Note 3, p. 94, Note 6, p. S60.
14. Carbo-phenothion	GC					Note 4, p. 27; Note 6, p. S73.
15. Chlordane	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
16. Chloro-propham	TLC					Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		6640 B			Note 3, p. 115; Note 4, p. 40.
18. 4,4,-DDD	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
19. 4,4,-DDE	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
20. 4,4,-DDT	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
21. Demeton-O	GC					Note 3, p. 25; Note 6, p. S51.
22. Demeton-S	GC					Note 3, p. 25; Note 6, p. S51.
23. Diazinon	GC					Note 3, p. 25; Note 4, p. 27; Note 6, p. S51.
24. Dicamba	GC					Note 3, p. 115.
25. Dichlofenthion	GC					Note 4, p. 27; Note 6, p. S73.
26. Dichloran	GC		6630 B & C			Note 3, p. 7.
27. Dicofol	GC				D3086–90, D5812–96(02)	
28. Dieldrin	GC	608	6630 B & C			Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
29. Dioxathion	GC					Note 4, p. 27; Note 6, p. S73.
30. Disulfoton	GC					Note 3, p. 25; Note 6 p. S51.
31. Diuron	TLC					Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	[5] 625	6410 B	6410 B–00		
33. Endosulfan II	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 8.
	GC/MS	[5] 625	6410 B	6410 B–00		
34. Endosulfan Sulfate	GC	608	6630 C			Note 8.
	GC/MS	625	6410 B	6410 B–00		
35. Endrin	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	[5] 625	6410 B	6410 B–00		
36. Endrin aldehyde	GC	608				Note 8.
	GC/MS	625				
37. Ethion	GC					Note 4, p. 27; Note 6, p. S73.
38. Fenuron	TLC					Note 3, p. 104; Note 6, p. S64.
39. Fenuron-TCA	TLC					Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
41. Heptachlor epoxide	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 6, p. S73; Note 8.
	GC/MS	625	6410 B	6410 B–00		
42. Isodrin	GC					Note 4, p. 27; Note 6, p. S73.
43. Linuron	GC					Note 3, p. 104; Note 6, p. S64.
44. Malathion	GC		6630 C			Note 3, p. 25; Note 4, p. 27; Note 6, p. S51.
45. Methiocarb	TLC					Note 3, p. 94; Note 6, p. S60.
46. Methoxychlor	GC		6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
47. Mexacarbate	TLC					Note 3, p. 94; Note 6, p. S60.
48. Mirex	GC		6630 B & C			Note 3, p. 7; Note 4, p. 27.
49. Monuron	TLC					Note 3, p. 104; Note 6, p. S64.
50. Monuron-TCA	TLC					Note 3, p. 104; Note 6, p. S64.
51. Nuburon	TLC					Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		6630 C			Note 3, p. 25; Note 4, p. 27.
53. Parathion ethyl	GC		6630 C			Note 3, p. 25; Note 4, p. 27.
54. PCNB	GC		6630 B & C			Note 3, p. 7.
55. Perthane	GC				D3086–90, D5812–96(02)	Note 4, p. 27.
56. Prometron	GC					Note 3, p. 83; Note 6, p. S68; Note 9.
57. Prometryn	GC					Note 3, p. 83; Note 6, p. S68; Note 9.
58. Propazine	GC					Note 3, p. 83; Note 6, p. S68; Note 9.
59. Propham	TLC					Note 3, p. 104; Note 6, p. S64.
60. Propoxur	TLC					Note 3, p. 94; Note 6, p. S60.
61. Secbumeton	TLC					Note 3, p. 83; Note 6, p. S68.
62. Siduron	TLC					Note 3, p. 104; Note 6, p. S64.
63. Simazine	GC					Note 3, p. 83; Note 6, p. S68; Note 9.
64. Strobane	GC		6630 B & C			Note 3, p. 7.
65. Swep	TLC					Note 3, p. 104; Note 6, p. S64.

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

Parameter	Method	EPA [2,7]	Standard methods 18th, 19th, 20th Ed.	Standard methods on-line	ASTM	Other
66. 2,4,5-T	GC		6640 B			Note 3, p. 115; Note 4, p. 40.
67. 2,4,5-TP (Silvex)	GC		6640 B			Note 3, p. 115; Note 4, p. 40.
68. Terbuthylazine GC						Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC	608	6630 B & C		D3086–90, D5812–96(02)	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B	6410 B–00		
70. Trifluralin	GC		6630 B			Note 3, p. 7; Note 9.

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 1C, 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 U.S. 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.

TABLE 1E.—LIST OF APPROVED RADIOLOGIC TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)				
		EPA [1]	Standard methods 18th, 19th, 20th ed.	Standard methods on-line	ASTM	USGS [2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter.	900.0	7110 B	7110 B–00	D1943–90, 96.	pp. 75 and 78[3].
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter.	Appendix B	7110 B	7110 B–00	D1943–90, 96.	p. 79.
3. Beta-Total, pCi per liter	Proportional counter	900.0	7110 B	7110 B–00	D1890–90, 96.	pp. 75 and 78[3].
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	7110 B–00	D1890–90, 96.	p. 79.
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500Ra B	7500–Ra B–01.	D2460–90, 97.	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500RaC	7500–RA C–01.	D3454–91, 97.	p. 81.

Table 1E notes:

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

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

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

* * * * *

TABLE IG.—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS (40 CFR PART 455)

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s)
8	Triadimefon	43121–43–3	507/633/525.1/1656
12	Dichlorvos	00062–73–7	1657/507/622/525.1
16	2,4-D; 2,4-D Salts and Esters [2,4-Dichlorophenoxyacetic acid]	00094–75–7	1658/515.1/615/515.2/555
17	2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid]	00094–82–6	1658/515.1/615/515.2/555
22	Mevinphos	07786–34–7	1657/507/622/525.1
25	Cyanazine	21725–46–2	629/507
26	Propachlor	01918–16–7	1656/508/608.1/525.1
27	MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid].	00094–74–6	1658/615/555
30	Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid].	00120–36–5	1658/515.1/615/515.2/555
31	MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid].	00093–65–2	1658/615/555
35	TCMTB [2-(Thiocyanomethylthio) benzothiazole]	21564–17–0	637
39	Pronamide	23950–58–5	525.1/507/633.1
41	Propanil	00709–98–8	632.1/1656
45	Metribuzin	21087–64–9	507/633/525.1/1656
52	Acephate	30560–19–1	1656/1657

TABLE IG.—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS (40 CFR PART 455)—Continued

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s)
53	Acifluorfen	50594–66–6	515.1/515.2/555
54	Alachlor	15972–60–8	505/507/645/525.1/1656
55	Aldicarb	00116–06–3	531.1
58	Ametryn	00834–12–8	507/619/525.1
60	Atrazine	01912–24–9	505/507/619/525.1/1656
62	Benomyl	17804–35–2	631
68	Bromacil; Bromacil Salts and Esters	00314–40–9	507/633/525.1/1656
69	Bromoxynil	01689–84–5	1625/1661
69	Bromoxynil octanoate	01689–99–2	1656
70	Butachlor	23184–66–9	507/645/525.1/1656
73	Captafol	02425–06–1	1656
75	Carbaryl [Sevin]	00063–25–2	531.1/632/553
76	Carbofuran	01563–66–2	531.1/632
80	Chloroneb	02675–77–6	1656/508/608.1/525.1
82	Chlorothalonil	01897–45–6	508/608.2/525.1/1656
84	Stirofos	00961–11–5	1657/507/622/525.1
86	Chlorpyrifos	02921–88–2	1657/508/622
90	Fenvalerate	51630–58–1	1660
103	Diazinon	00333–41–5	1657/507/614/622/525.1
107	Parathion methyl	00298–00–0	1657/614/622
110	DCPA [Dimethyl 2,3,5,6-tetrachloroterephthalate]	01861–32–1	508/608.2/525.1/515.1/515.2/1656
112	Dinoseb	00088–85–7	1658/515.1/615/515.2/555
113	Dioxathion	00078–34–2	1657/614.1
118	Nabonate [Disodium cyanodithioimidocarbonate]	00138–93–2	630.1
119	Diuron	00330–54–1	632/553
123	Endothall	00145–73–3	548/548.1
124	Endrin	00072–20–8	1656/505/508/608/617/525.1
125	Ethalfluralin	55283–68–6	[1]1656/[1]627
126	Ethion	00563–12–2	1657/614/614.1
127	Ethoprop	13194–48–4	1657/507/622/525.1
132	Fenarimol	60168–88–9	507/633.1/525.1/1656
133	Fenthion	00055–38–9	1657/622
138	Glyphosate [N-(Phossphonomethyl) glycine]	01071–83–6	547
140	Heptachlor	00076–44–8	1656/505/508/608/617/525.1
144	Isopropalin	33820–53–0	1656/627
148	Linuron	00330–55–2	553/632
150	Malathion	00121–75–5	1657/614
154	Methamidophos	10265–92–6	1657
156	Methomyl	16752–77–5	531.1/632
158	Methoxychlor	00072–43–5	1656/505/508/608.2/617/525.1
172	Nabam	00142–59–6	630/630.1
173	Naled	00300–76–5	1657/622
175	Norflurazon	27314–13–2	507/645/525.1/1656
178	Benfluralin	01861–40–1	[1]1656/[1]627
182	Fensulfothion	00115–90–2	1657/622
183	Disulfoton	00298–04–4	1657/507/614/622/525.1
185	Phosmet	00732–11–6	1657/622.1
186	Azinphos Methyl	00086–50–0	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	00056–38–2	1657/614
204	Pendimethalin	40487–42–1	1656
205	Pentachloronitrobenzene	00082–68–8	1656/608.1/617
206	Pentachlorophenol	00087–86–5	625/1625/515.2/555/515.1/525.1
208	Permethrin	52645–53–1	608.2/508/525.1/1656/1660
212	Phorate	00298–02–2	1657/622
218	Busan 85 [Potassium dimethyldithiocarbamate]	00128–03–0	630/630.1
219	Busan 40 [Potassium N-hydroxymethyl-N-methyldithiocarbamate]	51026–28–9	630/630.1
220	KN Methyl [Potassium N-methyldithiocarbamate]	00137–41–7	630/630.1
223	Prometon	01610–18–0	507/619/525.1
224	Prometryn	07287–19–6	507/619/525.1
226	Propazine	00139–40–2	507/619/525.1/1656
230	Pyrethrin I	00121–21–1	1660
232	Pyrethrin II	00121–29–9	1660
236	DEF [S,S,S-Tributyl phosphorotrithioate]	00078–48–8	1657
239	Simazine	00122–34–9	505/507/619/525.1/1656
241	Carbam-S [Sodium dimethyldithiocarbanate]	00128–04–1	630/630.1
243	Vapam [Sodium methyldithiocarbamate]	00137–42–8	630/630.1
252	Tebuthiuron	34014–18–1	507/525.1
254	Terbacil	05902–51–2	507/633/525.1/1656
255	Terbufos	13071–79–9	1657/507/614.1/525.1
256	Terbuthylazine	05915–41–3	619/1656
257	Terbutryn	00886–50–0	507/619/525.1
259	Dazomet	00533–74–4	630/630.1/1659
262	Toxaphene	08001–35–2	1656/505/508/608/617/525.1
263	Merphos [Tributyl phosphorotrithioate]	00150–50–5	1657/507/525.1/622
264	Trifluralin	01582–09–8	1656/508/617/627/525.1
268	Ziram [Zinc dimethyldithiocarbamate]	00137–30–4	630/630.1

---

[1] Monitor and report as total Trifluralin.

(b) * * *

REFERENCES, SOURCES, COSTS, AND TABLE CITATIONS:

* * * * *

(6) American Public Health Association. 1992, 1995, and 1998. Standard Methods for the Examination of Water and Wastewater. 18th, 19th, and 20th Edition (respectively). Available from: American Public Health Association, 1015 15th Street, NW., Washington, DC 20005. Standard Methods On-Line are available through the Standard Methods Web site (*www.standardmethods.org*). Tables IA, IB, IC, ID, IE.

* * * * *

(10) ASTM International. Annual Book of ASTM Standards, Water, and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994, 1996, 1999, Volume 11.02, 2000, and individual standards published after 2000. Available from: ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959, or *www.astm.org.* Tables IA, IB, IC, ID, and IE.

* * * * *

(17) AOAC—International. Official Methods of Analysis of AOAC—International, 16th Edition, (1995). Available from: AOAC—International, 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877. Table 1B, Note 3.

* * * * *

(63) Waters Corporation. 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, 508/482–2131 (Office), 508/482–3625 (FAX). Table IB, Note 54.

(64) Kelada–01, “Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate,’’ EPA 821–B–01–009 is available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. Phone: 800–553–6847. Table IB, Note 55.

(65) 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 Rd., Milwaukee, WI 53218, USA. Phone: 414–358–4200. Table IB, Note 56.

(66) “Methods for the Determination of Metals in Environmental Samples,’’ Supplement I, National Exposure Risk Laboratory—Cincinnati (NERL–CI), EPA/600/R–94/11, May 1994; and “Methods for the Determination of Inorganic Substances in Environmental Samples,’’ NERL–CI, EPA/600/R–93/100, August 1993 are available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800–553–6847. Table IB.

(67) “Determination of Inorganic Ions in Drinking Water by Ion Chromatography,’’ Rev. 1.0, 1997 is available from from *http://www.epa.gov/safetwater/methods/met300.pdf.* Table IB.

(68) Table IG Methods are available in “Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume I,’’ EPA 821–R–93–010A (August 1993, Revision I) and “Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume I,’’ EPA 821–R–93–010B (August 1993) are available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800–553–6847. Table IB.

(69) “Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry’’ [December 2003]. Available at *http://www.epa.gov/waterscience/methods/.*

* * * * *

(c) Under certain circumstances, the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon recommendation of the Alternate Test Procedure Program Coordinator, Washington, DC.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Alternate Test Procedure Program Coordinator, Washington, DC, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters are cited in Tables IA, IB, IC, ID, IE, IF, and IG are prescribed in Table II. Information in the table takes precedence over information in specific methods or elsewhere. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by the Regional Administrator to the Alternate Test Procedure Program Coordinator, Washington, DC, for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Alternate Test Procedure Program Coordinator, the Regional Administrator may grant a variance applicable to the specific discharge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.?≤

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

Parameter no./name	Container[1]	Preservation[2, 3, 17]	Maximum holding time[4, 17]
Table IA—Bacteria Tests:			
1–5 Coliform, total, fecal, and E. coli.	PA,G	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$[5]	6 hours.
6 Fecal streptococci	PA,G	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$5	do.
7 enterococci	PA,G	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$[5]	do.
Table IA—Protozoa Tests:			
8 Cryptosporidium	LDPE	≤6.00 °C [18]	96 hours.

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

Parameter no./name	Container[1]	Preservation[2, 3, 17]	Maximum holding time[4, 17]
9 Giardia	LDPE	≤6.00 °C [18]	96 hours.
Table IA—Aquatic Toxicity Tests:			
6–10 Toxicity, acute and chronic	P,G	Cool, ≤6.00 °C \16 18\	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P,G	Cool, ≤6.00 °C [18]	14 days.
2. Alkalinity	P,G	do	do.
4. Ammonia	P,G	Cool, ≤6.00 °C [18], H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P,G	Cool, ≤6.00 °C [18]	48 hours.
10. Boron	P or Quartz	HNO_3 to pH<2	6 months.
11. Bromide	P,G	none required	28 days.
14. Biochemical oxygen demand, carbonaceous.	P,G	Cool, ≤6.00 °C [18]	48 hours.
15. Chemical oxygen demand	P,G	Cool, ≤6.00 °C [18], H_2SO_4 to pH<2	28 days.
16. Chloride	P,G	none required	do.
17. Chlorine, total residual	P,G	do	Analyze within 15 minutes.
21. Color	P,G	Cool, ≤6.00 °C [18]	48 hours.
23–24. Cyanide, total and available (or CATC).	P,G	Cool, ≤6.00 °C [18], NaOH to pH<12, reducing agent [5]	14 days[6].
25. Fluoride	P	None required	28 days.
27. Hardness	P,G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P,G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P,G	Cool, ≤6.00 °C [18], H_2SO_4 to pH<2	28 days.
Metals[7]			
18. Chromium VI [7]	P,G	Cool, ≤6.00 °C [18], pH = 9.3–9.7 [21]	do.
35. Mercury (CVAA)	P,G	HNO_3 to pH<2	do.
35. Mercury (CVAFS) [17]	FP, G; and FP-lined cap[17].	5 mL/L 12N HCl or 5 mL/L BrCl [17]	28 days[17].
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI and mercury[7].	P,G	HNO_3 to pH<2 at least 24 hours prior to analysis.[20]	6 months.
38. Nitrate	P,G	Cool, ≤6.00 °C [18]	48 hours.
39. Nitrate-nitrite	P,G	Cool, ≤6.00 °C [18], H_2SO_4 to pH<2	28 days.
40. Nitrite	P,G	Cool, ≤6.00 °C [18]	48 hours.
41. Oil and grease	G	Cool to ≤6.00 °C [18], HCl or H_2SO_4 to pH<2	28 days.
42. Organic Carbon	P,G	Cool to ≤6.00 °C [18], HCl or H_2SO_4 or H_3PO_4, to pH<2	do.
44. Orthophosphate	P,G	Filter within 15 minutes of collection, Cool, ≤6.00 °C [18]	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, ≤6.00 °C [18], H_2SO_4 to pH<2	28 days.
49. Phosphorous (elemental)	G	Cool, ≤6.00 °C [18]	48 hours.
50. Phosphorous, total	P,G	Cool, ≤6.00 °C [18], H_2SO_4 to pH<2	28 days.
53. Residue, total	P,G	Cool, ≤6.00 °C [18]	7 days.
54. Residue, Filterable	P,G	do	do.
55. Residue, Nonfilterable (TSS)	P,G	do	do.
56. Residue, Settleable	P,G	do	48 hours.
57. Residue, Volatile	P,G	do	7 days.
61. Silica	P or Quartz	Cool, ≤6.00 °C [18]	28 days.
64. Specific conductance	P,G	do	do.
65. Sulfate	P,G	do	do.
66. Sulfide	P,G	Cool, ≤6.00 °C [18] add zinc acetate plus sodium hydroxide to pH>9	7 days.
67. Sulfite	P,G	None required	Analyze within 15 minutes.
68. Surfactants	P,G	Cool, ≤6.00 °C [18]	48 hours.
69. Temperature	P,G	None required	Analyze.
73. Turbidity	P,G	Cool, ≤6.00 °C [18]	48 hours.
Table IC.—Organic Tests.[8]			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$ [5]	14 days.
6,57, 106. Purgeable aromatic hydrocarbons.	do	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$ [5], HCl to pH 2[9]	do.
3,4, Acrolein and acrylonitrile	do	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$ [5], adjust pH to 4–5 [10]	do.
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols[11].	G, Teflon-lined cap	Cool, ≤6.00 °C [18], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction.
7, 38. Benzidines[11]	do	do	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters.[11].	do	Cool, ≤6.00 °C [18]	7 days until extraction, 40 days after extraction.
82–84. Nitrosamines[11, thnsp;14]	do	Cool, ≤6.00 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5]	do.
88–94. PCBs[11]	do	Cool, ≤6.00 °C [18]	1 year.
54, 55, 75, 79. Nitroaromatics and isophorone[11].	do	Cool, ≤6.00 °C [18] store in dark, 0.008% $Na_2S_2O_3$ [5]	do.

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

Parameter no./name	Container[1]	Preservation[2, 3, 17]	Maximum holding time[4, 17]
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons[11].	do	do	do.
15, 16, 21, 31, 87. Haloethers[11]	do	Cool, ≤6.00 °C[18], 0.008% $Na_2S_2O_3$[5]	do.
29, 35–37, 63–65, 107. Chlorinated hydrocarbons.[11].	do	Cool, ≤6.00 °C[18]	do.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs.[11].			
Aqueous Samples: Field and Lab Preservation.	G	Cool, ≤6.00 °C[18], 0.008% $Na_2S_2O_3$[5], pH<9	1 year.
Solids & Mixed Phase Samples: Field Preservation.	do	Cool, ≤6.00 °C[18]	7 days.
Tissue Samples: Field Preservation.	do	Cool, ≤6.00 °C[18]	24 hours.
Solids, Mixed Phase, and Tissue Samples: Lab Preservation.	do	Freeze, ≤ −10 °C	1 year.
Table ID-Pesticides Tests:			
1–70. Pesticides.[11]	do	Cool, ≤6.00 °C[18], pH 5–9[15]	do.
Table IE-Radiological Tests:			
1–5. Alpha, beta and radium	P,G	HNO_3 to pH<2	6 months.

[1] "P" is polyethylene or polytetrafluoroethylene (PTFE; Teflon), or other fluoropolymer, unless otherwise stated in this Table II. "FP" is fluoropolymer. "G" is glass. "PA" is any plastic that is made of a sterlizable 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 to be collected automatically over time (*e.g.*, using a 24-hour composite sampler; see 40 CFR 122.21(g)(7) and 40 CFR part 403, Appendix E), refrigerate the sample at ≤6.00 °C during collection unless otherwise specified in this Table II or in the method(s). For a composite sample collected automatically over time to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6.00 °C unless otherwise specified 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 any of the aliquots split from a composite sample collected automatically over time; otherwise, preserve the sample or aliquots within 15 minutes of collection. If a composite measurement is required but a composite sample collected automatically over time 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-hr. 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 the 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 through the United States mails, 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 requirements 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 six 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 § 136.3(e). For a grab sample, the holding time begins at the time of collection for a composite sample collected automatically over time (*e.g.*, using a 24-hour composite sample; see 40 CFR 122.21(g)(7) and 40 CFR part 403, Appendix E), the holding time begins at the time of the *end* of collection of the composite samples. 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 they know it is necessary to maintain sample stability. See § 136.3(e) for details.

[5] Add ascorbic acid or sodium borohydride ($NaBH_4$) reagent if (and only if) oxidants (*e.g.*, chlorine) are present. Add enough reagent to reduce any oxidants that are present. Generally, 0.1 g/L $NaBH_4$ can reduce 50 mg/L of chlorine (see method "Kelada-01" for more information). Methods recommending ascorbic acid generally specify to increase ascorbic acid in 0.6 mg/L increments until oxidants are removed. After adding reagent, test the sample using KI paper or a chlorine/oxidant test method to make sure all chlorine/oxidant is removed. If chlorine/oxidant remains, add more reagent. Do not add excess reagent, however, because this may interfere with test results.

[6] Collect the sample in an amber glass bottle with PTFE-lined cap. Immediately after collection, preserve the sample using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to ≥12 by addition of sodium hydroxide and refrigeration as specified:

(1) Sulfide: The maximum holding time for an untreated sample is 24 hours when sulfide is present. Optionally, the sample may be treated and the maximum holding time extended to 14 days. Generally, the laboratory should test the sample with lead acetate test paper to determine the presence or absence of sulfide ion. However, for cyanide methods using amperometric detection systems (*e.g.*, OIA–1677 for available cyanide), sulfide levels below those detectable with lead acetate paper (approximately 5 ppm) may produce a false positive signal for cyanide. If there is reason to suspect sulfide levels below the detectable level of lead acetate paper when using an amperometric method, test the sample using a more sensitive sulfide method to determine if the treatment (described below) is required. If sulfide ion is present, treat the sample immediately (within 15 minutes of collection) with sufficient solid lead carbonate to remove sulfide (as evidenced by a lead acetate test paper), and immediately filter into another sample bottle to remove precipitated lead sulfide. If sulfide ion is suspected to be present, but its presence is not detected by the lead acetate paper test, two samples must be collected. One is treated for the presence of sulfide and immediately filtered, while the second is not treated for sulfide. Analyze both samples and report the lower of the two results.

(2) Sulfide and particulate matter: If the sample contains sulfide and particulate matter that would be removed by filtration, filter the sample prior to treatment with lead carbonate to assure that cyanides associated with the particulate matter are included in the measurement. Save the particulate matter and treat the filtrate using the sulfide removal procedure above. Combine and homogenize the collected particulate and treated filtrate prior to shipment to the laboratory for analysis.

(3) Aldehydes: Treat samples known or suspected to contain formaldehyde, acetaldehyde, or other water soluble aldehydes with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(4) Chlorine, hypochlorite, or other oxidants: Treat samples known or suspected to contain chlorine, hypochlorite, or other oxidants as directed in footnote 5. EPA Method 330.4 or 330.5 may be used for the measurement of residual chlorine.

[7] For dissolved metals, filter the sample within 15 minutes of collection and before adding preservatives.

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

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

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

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (*i.e.*, use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6.00 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

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

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

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

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

[16] Sufficient ice should be placed 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, it is necessary to immediately measure the temperature of the samples and confirm that the 6.00 °C 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.

[17] Samples collected for the determination of trace level mercury (100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipment, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that have been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[18] Samples must be preserved at ≤6.00 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample analysis is maintained on file and accepted as valid by the permitting authority. Also, for the purposes of NPDES monitoring, the specification of "≤6.00 °C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods.

[19] "do" means ditto, or same as the entry immediately above this column.

[20] Samples can be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls.

[21] To achieve the 28 day holding time, use sodium hydroxide and the ammonium sulfate buffer solution specified in EPA Method 218.6.

3. Section 136.4 is amended by revising the first sentence of paragraph (d) introductory text to read as follows:

§ 136.4 Application for alternate test procedures.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Alternate Test Procedure Program Coordinator, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460. * * *

4. Section 136.5 is amended:

a. In paragraph (b) by revising the second sentence.

b. By revising paragraph (c).

c. In paragraph (d) by revising the second and third sentences.

d. By revising paragraphs (e)(1) and (e)(2).

The revisions read as follows:

§ 136.5 Approval of alternate test procedures.

* * * * *

(b) * * * Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application and shall forward this decision to the Director of the State Permit Program and to the Alternate Test Procedure Program Coordinator, Washington, DC.

(c) Before approving any application for an alternate test procedure proposed by the responsible person or firm making the discharge, the Regional Administrator shall forward a copy of the application to the Alternate Test Procedure Program Coordinator, Washington, DC.

(d) * * * Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Alternate Test Procedure Program Coordinator, Washington, DC. A copy of all approval and rejection notifications will be forwarded to the Alternate Test Procedure Program Coordinator, Washington, DC, for the purposes of national coordination.

(e) *Approval for nationwide use.* (1) As expeditiously as is practicable after receipt by the Alternate Test Procedure Program Coordinator, Washington, DC, of an application for an alternate test procedure for nationwide use, the Alternate Test Procedure Program Coordinator, Washington, DC, shall notify the applicant in writing whether the application is complete. If the application is incomplete, the applicant shall be informed of the information necessary to make the application complete.

(2) As expeditiously as is practicable after receipt of a complete package, the Alternate Test Procedure Program Coordinator shall perform any analysis necessary to determine whether the alternate test procedure satisfies the applicable requirements of this part, and the Alternate Test Procedure Program Coordinator shall recommend to the Administrator that he/she approve or reject the application and shall also notify the application of the recommendation.

* * * * *

5. Section 136.6 is added to part 136 to read as follows:

§ 136.6 Method modifications and analytical requirements.

(a) *Definitions.* As 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.*, colorimetery, mass spectrometry).

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

(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 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) *Allowable changes.* Except as set forth in paragraph (b)(3) of this section, an Analyst may modify a test procedure (analytical method) provided that the chemistry of the method or the determinative technique is not changed, and provided that the requirements of paragraph (b)(2) of this section are met.

(i) Potentially allowable modifications regardless of current method performance include changes between automated and manual discrete instrumentation; changes in the calibration range (provided that the modified range covers any relevant regulatory limit); changes in equipment such as using similar equipment from a vendor other than that mentioned in the method (*e.g.*, a purge-and-trap device from OIA rather than Tekmar), changes in equipment operating parameters such as changing the monitoring wavelength of a colorimeter or modifying the temperature program for a specific GC column; changes to chromatographic columns (treated in greater deal in paragraph (d) of this section); and increases in purge-and-trap sample volumes (provided specifications in paragraph (e) of this section are met). The changes are only allowed provided that all the requirements of paragraph (b)(2) of this section are met.

(ii) 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 as specified in *Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring* (EPA 821–B–93–001, June 1993), 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.

(iii) If the characteristics of a wastewater matrix result in poor sample dispersion or reagent deposition on equipment and prevents the Analyst from meeting QC requirements, the Analysts may attempt to resolve the issue by adding an inert surfactant (*i.e.* a surfactant that will not affect the Chemistry of the Method), which may include 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)(2) of this section are met. Chlorinated samples must be dechlorinated prior to the addition of such surfactant.

(2) *Requirements.* A modified method must produce Equivalent Performance for the analyte(s) of interest, and the Equivalent Performance must be documented.

(i) *Requirements for Establishing Performance.*

(A) If the approved method contains QC tests and QC acceptance criteria, the QC tests must be used with the modified method and the QC acceptance criteria must be met. 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 (*i.e.*, as 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 paragraph (b)(2)(i)(A) of this section, the analyst must employ QC tests specified in *Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA–821–B–98–002; March 1999) and meet the QC provisions specified therein. In addition, the Analyst must perform on-going 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 of a discharge analyzed), and analysis of an ongoing precision and recovery sample and a blank with each batch of 20 or fewer samples.

(C) Calibration must be performed using the modified method and the modified method must be tested with every wastewater matrix to which it will be applied (up to nine distinct matrices; as described in the ATP Protocol, after validation in nine distinct matrices, the method may be applied to all wastewater 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.

(D) Analysts are obligated to test representative effluents. In addition, the non-modified approved method may be required to resolve any controversies.

(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. 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.* This paragraph does not apply to a method for a method-defined analyte or a change that would result in measurement of a different form or species of an analyte (*e.g.*, a change to a metals digestion or total cyanide distillation). This paragraph (b)(3) also does not apply to changes in sample preservation and/or holding time.

(c) *Analytical requirements for multi-analyte methods (target analytes).* For the purpose of NPDES reporting, the discharger or permittee must meet QC requirements only for the analyte(s) being measured and reported under the NPDES permit.

(d) *Capillary column.* Use of a capillary (open tubular) GC column with EPA Methods 601–613, 624, 625, and 1624B in appendix A to this part is allowed provided that all QC tests in the method are performed and all QC acceptance criteria are met. When changing from a packed column to a capillary column, the analyst must establish a new record of analyte retention times and keep these on file along with other startup test and ongoing QC data.

(e) *Increased sample volume in purge and trap methodology.* Increased sample volumes, up to a maximum of 25 mL, are allowed provided that the height of the water column in the purge vessel is at least 5 cm. The analyst should also use one or more surrogate analytes that are chemically similar to the analytes of interest in order to demonstrate that the increased sample volume does not adversely affect the analytical results.

6. Section 136.7 is added to part 136 to read as follows:

§ 136.7 Reporting.

(a) Demonstration of compliance with a permit must be based upon testing which meets QC requirements in this part, including QC requirements in the method used for the testing.

(b) Failure to meet the QC requirements in this part, including QC requirements in the approved method, does not relieve a discharger or permittee of timely reporting of test results.

(c) Results from tests must be reported to the level specified in the analytical method or permit, whichever is lower.

(d) Where a conflict is created between reporting requirements in this section and the reporting requirements in an analytical method listed in this part, reporting requirements in this section supersede reporting requirements in the analytical method.

Appendices C and D [Removed]

7. Appendices C and D to part 136 are removed.

PART 141—NATIONAL PRIMARY DRINKING WATER REGULATIONS

1. The authority citation for part 141 continues to read as follows:

Authority: 42 U.S.C. 300f, 300g–1, 300g–2 300g–3, 300g–4, 300g–5, 300g–6, 300j–4, 300j–9, and 300j–11.

2. Section 141.21 is amended by adding a sentence to the end of footnote 1 to the Table in paragraph (f)(3) to read as follows:

§ 141.21 Coliform sampling.

* * * * *

(f) * * *

(3) * * *

[1] * * * In addition, the following on-line versions with the noted approval date may also be used: 2310 B–01, 9215 B–00, 9221 A, B, D–99, 9222 A, B, C–97, and 9223–97.

* * * * *

3. Section 141.23 is amended:

a. In paragraph (a)(4)(i) by revising the entries for “Cyanide,’’ “Nitrate,’’ and “Nitrite’’ in the table, and by adding a new footnote 9 to the table.

b. In paragraph (k)(1) by revising the table. The revisions and addition read as follows:

§ 141.23 Inorganic chemical sampling and analytical requirements.

* * * * *

(a) * * *

(4) * * *

(i) * * *

DETECTION LIMITS FOR INORGANIC CONTAMINANTS

Contaminant	MCL (mg/L)	Methodology	Detection Limit (mg/L)
* * * * * * *			
Cyanide	0.2	Distillation, Spectrophotometric [3]	0.02
		Distillation, Automated, Spectrophotometric [3]	0.005
		Distillation, Amenable, Spectrophotometric [4]	0.02
		Distillation, Selective Electrode [3]	0.05

DETECTION LIMITS FOR INORGANIC CONTAMINANTS—Continued

Contaminant	MCL (mg/L)	Methodology	Detection Limit (mg/L)
		UV, Distillation, Spectrophotometric[9]	0.0005
		Distillation, Spectrophotometric[3]	0.0006
		Ligand Exchange with Amperometry[4]	0.0005

* * * * * * *

Contaminant	MCL (mg/L)	Methodology	Detection Limit (mg/L)
Nitrate	10 (as N)	Manual Cadmium Reduction	0.01
		Automated Hydrazine Reduction	0.01
		Automated Cadmium Reduction	0.05
		Ion Selective Electrode	1
		Ion Chromatography	0.01
		Capillary Ion Electrophoresis	0.076
Nitrite	1 (as N)	Spectrophotometric	0.01
		Automated Cadmium Reduction	0.05
		Manual Cadmium Reduction	0.01
		Ion Chromatography	0.004
		Capillary Ion Electrophoresis	0.103

* * * * * * *

---

* * * * *

[3] Screening method for total cyanides.

[4] Measures "free" cyanides.

* * * * *

[9] Measures total cyanides when UV-digestor is used, and "free" cyanides when UV-digestor is bypassed.

* * * * *

(k) * * *

(1) * * *

Contaminant	Methodology[13]	EPA	ASTM[3]	SM[4] (18th, 19th ed.)	SM[4] (20th ed.)	SM on-line[22]	Other
1. Alkalinity	Titrimetric		D1067–92, 02 B	2320 B	2320 B	2320 B–97	
	Electrometric titration						I–030–85[5]
2. Antimony	Inductively Coupled Plasma (ICP)—Mass Spectrometry.	200.8[2].					
	Hydride-Atomic Absorption.		D3697–92				
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.			3113 B		3113 B–99	
3. Arsenic[14]	Inductively Coupled Plasma[15].	200.7[2]		3120 B	3120 B	3120 B–99	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption Furnace.		D2972–97, 03 C	3113 B		3113 B–99.	
	Hydride Atomic Absorption.		D2972–97, 03 B	3114 B		3114 B–97	
4. Asbestos	Transmission Electron Microscopy.	100.1[9]					
	Transmission Electron Microscopy.	100.2[10]					
5. Barium	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Direct			3111 D		3111 D–99	
	Atomic Absorption; Furnace.			3113 B		3113 B–99	
6. Beryllium	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.		D3645–97, 03 B	3113 B	3113 B–99		
7. Cadmium	Inductively Coupled Plasma.	200.7[2]					
	ICP-Mass Spectrometry	200.8[2].					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.			3113 B		3113 B–99	
8. Calcium	EDTA titrimetric		D511–93, 03 A	3500–Ca D	3500–Ca B	3500–Ca B–97	
	Atomic Absorption; Direct Aspiration.		D511–93, 03 B	3111 B		3111 B–99	
	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	

Contaminant	Methodology[13]	EPA	ASTM[3]	SM[4] (18th, 19th ed.)	SM[4] (20th ed.)	SM on-line[22]	Other
	Ion Chromatography		D6919–03				
9. Chromium	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.			3113 B		3113 B–99	
10. Copper	Atomic Absorption; Furnace.		D1688–95, 02 C	3113 B		3113 B–99	
	Atomic Absorption; Direct Aspiration.		D1688–95, 02 A	3111 B		3111 B–99	
	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	
	ICP-Mass spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
11. Conductivity	Conductance	D1125–95 (99) A.	2510 B	2510 B	2510 B–97	.	
12. Cyanide	Manual Distillation followed by.		D2036–98A	4500–CN C	4500–CN–C		
	Spectro-photometric, Amenable.		D2036–98B	4500–CN G	4500–CN G	4500–CN G–99	
	Spectro-photometric Manual.		D2036–98A	4500–CN E	4500–CN E	4500–CN E99	I–3300–85[5]
	Spectro-photometric Semi-automated.	335.4[6]					
	Selective Electrode			4500–CN F	4500–CN F	4500–CN F–99	
	UV, Distillation, Spectro-photometric.						Kelada–01[17]
	Distillation, Spectro-photometric.						QuikChem 10–204–00–1–X[18]
	Ligand Exchange [and Amperometry[21].		D6888–03				OIA–1677, DW[20]
13. Fluoride	Ion Chromatography	300.0[6]	D4327–97, 03	4110 B	4110 B	4110 B–00	
	Manual Distill.; Color. SPADNS.			4500–F B, D	4500–F B, D	4500–F B, D–97.	
	Manual Electrode		D1179–93, 99 B	4500–F C	4500–F C	4500–F C–97	
	Automated Electrode						380–75WE[11]
	Automated Alizarin			4500–F E	4500–F E	4500–F E–97	129–71W[11]
	Capillary Ion Electrophoresis.						D6508, Rev. 2[19]
14. Lead	Atomic Absorption Furnace.		D3559–96, 03 D	3113 B		3113 B–99	
	ICP-Mass spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Differential Pulse Anodic Stripping Voltametry.						Method 1001[16]
15. Magnesium	Atomic Absorption		D511–93, 03 B	3111 B		3111 B–99	
	ICP	200.7[2]		3120 B	3120 B	3120 B–99	
	Complexation Titrimetric Methods.		D511–93, 03 A	3500–Mg E	3500–Mg B	3500–Mg B–97	
	Ion Chromatography		D6919–03				
16. Mercury	Manual, Cold Vapor	245.1[2]	D3223–97, 02	3112 B		3112 B–99.	
	Automated, Cold Vapor	245.2[1]					
	ICP-Mass Spectrometry	200.8[2]					
17. Nickel	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Direct			3111 B		3111 B–99	
	Atomic Absorption; Furnace.			3113		3113 B–99	
18. Nitrate	Ion Chromatography	300.0[6]	D4327–97, 03	4110 B	4110 B	4110 B–00	B–1011[8]
	Automated Cadmium Reduction.	353.2[6]	D3867–90 A	4500–NO_3 F	4500–No_3 F	4500–NO_3 F–00.	
	Ion Selective Electrode			4500–NO_3 D	4500–NO_3 D	4500–NO_3 D–00.	601[7]
	Manual Cadmium Reduction.		D3867–90 B	4500–NO_3 E	4500–NO_3 E	4500–NO_3 E–00.	
	Capillary Ion Electrophoresis.						D6508, Rev. 2[19]
19. Nitrite	Ion Chromatography	300.0[6]	D4327–97	4110 B	4110 B	4110 B–00	B–1011[8]
	Automated Cadmium Reduction.	353.2[6]	D3867–90 A	4500–NO_3 F	4500–NO_3 F	4500–NO_3 F–00.	
	Manual Cadmium Reduction.		D3867–90 B	4500–NO_3 E	4500–NO_3 E	4500–NO_3 E–00.	
	Spectro-photometric			4500–NO_2 B	4500–NO_2 B	4500–NO_2 B–00.	

Contaminant	Methodology[13]	EPA	ASTM[3]	SM[4] (18th, 19th ed.)	SM[4] (20th ed.)	SM on-line[22]	Other
	Capillary Ion Electrophoresis.						D6508, Rev. 2[19]
20. Ortho-phosphate[12].	Colorimetric, Automated, Ascorbic Acid.	365.1[6]		4500–P F	4500–P F		
	Colorimetric, ascorbic acid, single reagent.		D515–88A	4500–P E	4500–P E		
	Colorimetric Phosphomolybdate;.						I–1601–85[5]
	Automated-segmented Flow;.						I–2601–90[5]
	Automated Discrete						I2598–85[5]
	Ion Chromatography	300.0[6]	D4327–97, 03	4110 B	4110 B	4110 B–00	
	Capillary Ion Electrophoresis.						D6508, Rev. 2[19]
21. pH	Electrometric	150.1, 150.2[1].	D1293–95, 99	$4500–H^+$ B	$4500–H^+$ B	$4500–H^+$B–00	
22. Selenium	Hydride-Atomic Absorption.		D3859–98, 03 A	3114 B		3114 B–97	
	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					
	Atomic Absorption; Furnace.		D3859–98, 03 B	3113 B		3113 B–99	
23. Silica	Colorimetric, Molybdate Blue;.						I–1700–85[5]
	Automated-segmented Flow.						I–2700–85[5]
	Colorimetric		D859–95, 00				
	Molybdosilicate			4500–Si D	4500–SiO_2 C	4500–SiO_2 C–97.	
	Heteropoly blue			4500–Si E	4500–SIO_2 D	4500–SiO_2 D–97.	
	Automated for Molybdate-reactive Silica.			4500–Si F	4500–SiO_2 E	4500–SiO_2 E–97.	
	Inductively Coupled Plasma.	200.7[2]		3120 B	3120 B	3120 B–99	
24. Sodium	Inductively Coupled Plasma.	200.7[2]					
	Atomic Absorption; Direct Aspiration.			3111 B		3111 B–99	
	Ion Chromatography		D6919–03				
25. Temperature	Thermometric			2550	2550	2550–00	
26. Thallium	ICP-Mass Spectrometry	200.8[2]					
	Atomic Absorption; Platform.	200.9[2]					

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of the following documents listed in footnotes 1–11, 16–20 and 22 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 below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, 1301 Constitution Avenue, NW., EPA West, Room B102, Washington, DC 20460 (Telephone: 202–566–2426); or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC 20408.

[1] "Methods for Chemical Analysis of Water and Wastes," EPA/600/4–79/020, March 1983. Available at NTIS, PB84–128677.

[2] "Methods for the Determination of Metals in Environmental Samples—Supplement I," EPA/600/R–94/111, May 1994. Available at NTIS, PB95–125472.

[3] *Annual Book of ASTM Standards,* 1994, 1996, 1999, or 2003, Vols. 11.01 and 11.02, ASTM International; any year containing the cited version of the method may be used. The previous versions of D1688–95A, D1688–95C (copper), D3559–95D (lead), D1293–95 (pH), D1125–91A (conductivity) and D859–94 (silica) are also approved. These previous versions D1688–90A, C; D3559–90D, D1293–84, D1125–91A and D859–88, respectively are located in the Annual Book of ASTM Standards, 1994, Vol. 11.01. Copies may be obtained from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[4] *Standard Methods for the Examination of Water and Wastewater,* 18th edition (1992), 19th edition (1995), or 20th edition (1998). American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. The cited methods published in any of these three editions may be used, except that the versions of 3111 B, 3111 D, 3113 B and 3114 B in the 20th edition may not be used.

[5] Method I–2601–90, Methods for Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments, Open File Report 93–125, 1993; For Methods I–1030–85; I–1601–85; I–1700–85; I–2598–85; I–2700–85; and I–3300–85 See Techniques of Water Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A–1, 3rd ed.,1989; Available from Information Services, U.S. Geological Survey, Federal Center, Box 25286, Denver, CO 80225–0425.

[6] "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA/600/R–93/100, August 1993. Available at NTIS, PB94–120821. Available at NTIS, PB94–120821, 5285 Port Royal Road, Springfield, VA 22161. The toll free telephone number is 800–553–6847.

[7] The procedure shall be done in accordance with the Technical Bulletin 601 "Standard Method of Test for Nitrate in Drinking Water," July 1994, PN 221890–001, Analytical Technology, Inc. Copies may be obtained from ATI Orion, 529 Main Street, Boston, MA 02129.

[8] Method B–1011, "Waters Test Method for Determination of Nitrite/Nitrate in Water Using Single Column Ion Chromatography," August 1987. Copies may be obtained from Waters Corporation, Technical Services Division, 34 Maple Street, Milford, MA 01757.

[9] Method 100.1, Analytical Method For Determination of Asbestos Fibers in Water," EPA/600/4–83/043, EPA, September 1983. Available at NTIS, PB83–260471.

[10] Method 100.2, "Determination of Asbestos Structure Over 10-um In Length In Drinking Water," EPA/600/R–94/134, June 1994. Available at NTIS, PB94–201902.

[11] Industrial Method No. 129–71W, "Fluoride in Water and Wastewater," December 1972, and Method No. 380–75WE, "Fluoride in Water and Wastewater," February 1976, Technicon Industrial Systems. Copies may be obtained from Bran & Luebbe, 1025 Busch Parkway, Buffalo Grove, IL 60089.

[12] Unfiltered, no digestion or hydrolysis.

[13] Because MDLs reported in EPA Methods 200.7 and 200.9 were determined using a 2X preconcentration step during sample digestion, MDLs determined when samples are analyzed by direct analysis (*i.e.,* no sample digestion) will be higher. For direct analysis of cadmium and arsenic by Method 200.7, and arsenic by Method 3120 B sample preconcentration using pneumatic nebulization may be required to achieve lower detection limits. Preconcentration may also be required for direct analysis of antimony, lead, and thallium by Method 200.9; antimony and lead by Method 3113 B; and lead by Method D3559–90D unless multiple in-furnace depositions are made.

[14] If ultrasonic nebulization is used in the determination of arsenic by Methods 200.7, 200.8, or SM 3120 B, the arsenic must be in the penta-valent state to provide uniform signal response. For methods 200.7 and 3120 B, both samples and standards must be diluted in the same mixed acid matrix concentration of nitric and hydrochloric acid with the addition of 100 mL of 30% hydrogen peroxide per 100 mL of solution. For direct analysis of arsenic with method 200.8 using ultrasonic nebulization, samples and standards must contain one mg/L of sodium hypochlorite.

[15] After January 23, 2006 analytical methods using the ICP–AES technology, may not be used because the detection limits for these methods are 0.008 mg/L or higher. This restriction means that the two ICP–AES methods (EPA Method 200.7 and SM 3120 B) approved for use for the MCL of 0.05 mg/L may not be used for compliance determinations for the revised MCL of 0.01 mg/L. However, prior to 2005 systems may have compliance samples analyzed with these less sensitive methods.

[16] The description for Method Number 1001 for lead is available from Palintest, LTD, 21 Kenton Lands Road, P.O. Box 18395, Erlanger, KY 41018. Or from the Hach Company, P.O. Box 389, Loveland, CO 80539.

[17] The description for the Kelada-01 Method, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, And Thiocyanate," Revision 1.2, August 2001, EPA #821–B–01–009 for cyanide is available from the National Technical Information Service (NTIS), PB 2001–108275, 5285 Port Royal Road, Springfield, VA 22161.

[18] The description for the QuikChem Method 10–204–00–1–X, "Digestion and distillation of total cyanide in drinking and wastewaters using MICRO DIST and determination of cyanide by flow injection analysis," Revision 2.1, November 30, 2000 for cyanide is available from Lachat Instruments, 6645 W. Mill Rd., Milwaukee, WI 53218, USA. Phone: 414–358–4200.

[19] 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, 508/482–2131 (Office), 508/482–3625 (FAX).

[20] Method OIA–1677, DW "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," January 2004. Available at NTIS, 5285 Port Royal Road, Springfield, VA 22161. The toll free telephone number is 800–553–6847.

[21] Sulfide levels below those detected using lead acetate paper may produce positive method interferences. Test samples using a more sensitive sulfide method to determine if a sulfide interference is present, and treat samples accordingly.

[22] Standard Methods On-line are available at *http://www.standardmethods.org.*

* * * * *

4. Section 141.24 is amended by revising the entries 23, 24, 26, 49, and 50 in the table in paragraph (e)(1) to read as follows:

§ 141.24 Organic chemicals, sampling and analytical requirements.

* * * * *

(e) * * *

(1) * * *

Contaminant	EPA method[1]	Standard methods	ASTM	Other
*	*	*	*	*
23. 2,4-D[4] (as acids, salts, and esters)	515.2, 555, 515.1, 515.3, 515.4	....	D5317–93, 98 (2003)	....
24. 2,4,5-TP[4] (Silvex)	515.2, 555, 515.1, 515.3, 515.4	....	D5317–93, 98 (2003)	....
*	*	*	*	*
26. Atrazine[2]	507, 525.2, 508.1, 505, 551.1	....	....	....
*	*	*	*	*
49. Pentachlorophenol	515.2, 525.2, 555, 515.1, 515.3, 515.4	....	D5317–93, 98 (2003)	....
50. Picloram[4]	515.2, 555, 515.1, 515.3, 515.4	....	D5317–93, 98 (2003)	....
*	*	*	*	*

[1] For previously approved EPA methods which remain available for compliance monitoring until June 1, 2001, see paragraph (e)(2) of this section.

[2] Substitution of the detector specified in 505, 507, 508, 508.1 for the purpose of achieving lower detection limits is allowed as follows. Either an electron capture or nitrogen phosphorus detector may be used provided all regulatory requirements and quality control criteria are met.

* * * * *

[4] Each community and non-transient non-community water system shall take four consecutive quarterly samples for each contaminant listed in § 141.61(a)(2) through (21) during each compliance period, beginning in the initial compliance period.

5. Section 141.25 is amended by revising the table in paragraph (a) to read as follows.

§ 141.25 Analytical methods for radioactivity.

(a) * * *

Contaminant	Methodology	Reference (method of page number)								
		EPA[1]	EPA[2]	EPA[3]	EPA[4]	SM[5]	ASTM[6]	USGS[7]	DOE[8]	Other
Naturally Occurring:										
Gross alpha and beta.[11]	Evaporation	900.0	p 1	00–01	p 1	302, 7110 B, 7110 B–00		R–1120–76		
Gross alpha[11]	Co-precipitation	....		00–02			7110 C, 7110 C–00			
Radium 226	Radon Emanation	903.1	p 16	Ra–04	p 19	305, 7500–Ra C, 7500 Ra C–01	D3454–97	R–1141–76	Ra–04	NY[9]
	Radio-chemical	903.0	p 13	Ra–03		304, 7500–Ra B, 7500–Ra B–01	D2460–97	R–1140–76		GA.[13]
Radium 228	Radio-chemical	904.0	p 24	Ra–05	p 19	7500–Ra D, 7500–Ra D–01		R–1142–76		NY[9], NJ[10] GA[13]
Uranium[12]	Radio-chemical	908.0				7500–U B, 7500–U B–00				
	Fluorometric	908.1				7500–U C (17th Ed.)	2907–97	R–1180–76, R–1181–76	U–04	
	Alpha Spectrometry	....		00–07	p 33	7500–U C (18th, 19th, or 20th Ed.), 7500–U–C–00	D3972–97, 02	R–1182–76	U–02	
	Laser Phos-phorometry.	....					D5174–97, 02			
Man-Made:										

Contaminant	Methodology	Reference (method of page number)								
		EPA[1]	EPA[2]	EPA[3]	EPA[4]	SM[5]	ASTM[6]	USGS[7]	DOE[8]	Other
Radioactivecesium	Radio-chemical	901.0	p 4			7500–Cs B, 7500–Cs B–02	D2459–72	R–1111–76		
	Gamma ray spectrometry.	901.1			p 92	7120, 7120–97	D3649–91, 98a	R–1110–76	4.5.2.3	
Radioactive iodine	Radio-chemical	902.0	p 6			7500–I B, 7500–I B–00				
	....	....	p 9			7500–I C, 7500–I C–00				
	....	....				7500–I D, 7500–I D–00	3649–91, 98a			
	Gamma ray spectrometry.	901.1			p 92	7120, 7120–97	D4785–93, 00a		4.5.2.3	
Radioactive Strontium 89, 90	Radio-chemical	905.0	p 29	Sr–04	p 65	303, 7500–Sr B 7500–Sr B–01		R–1160–76	Sr–01, Sr–02	
Tritium	Liquid Scintillation	906.0	p 34	H–02	p 87	306, 7500–3H B, 7500–3H B–00	D4107–91, 98 (2002)	R–1171–76		
Gamma Emitters	Gamma Ray	901.1			p 92	7120, 7120–97	D3649–91, 98a	R–1110–76	Ga–01–R	
	Spectrometry	902.0				7500–Cs B, 7500–Cs B–02	D4785–93, 00a			
	....	901.0				7500–I B, 7500–I B–00				

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of documents 1 through 10 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 below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, EPA West, 1301 Constitution Avenue, NW., Room B102 , Washington, DC 20460 (Telephone: 202–566–2426); or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[1] "Prescribed Procedures for the Measurement of Radioactivity in Drinking Water.'' EPA 600/4–80–032, August 1980. Available at the U.S. Department of Commerce, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 (Telephone 800–553–6847), PB 80–224744.

[2] "Interim Radiochemical Methodology for Drinking Water,'' EPA 600/4–75–008(revised), March 1976. Available NTIS, 5285 Port Royal Road, Springfield, VA 22161 (Telephone 800–553–6847), PB 253258.

[3] "Radiochemistry Procedures Manual,'' EPA 520/5–84–006, December, 1987. Available NTIS, 5285 Port Royal Road, Springfield, VA 22161 (Telephone 800–553–6847), PB 84–215581.

[4] "Radiochemical Analytical Procedures for Analysis of Environmental Samples,'' EMSL LV 053917, March 1979. Available at NTIS, 5285 Port Royal Road, Springfield, VA 22161 (Telephone 800–553–6847).

[5] "Standard Methods for the Examination of Water and Wastewater,'' 13th, 17th, 18th, 19th Editions, or 20th edition, 1971, 1989, 1992, 1995, 1998. Available at American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20005 . Methods 302, 303, 304, 305 and 306 are only in the 13th edition. Methods 7110B, 7500–Ra B, 7500–Ra C, 7500–Ra D, 7500–U B, 7500–Cs B, 7500–I B, 7500–I C, 7500–I D, 7500–Sr B, 7500–3H B are in the 17th, 18th, 19th and 20th editions. Method 7110 C is in the 18th, 19th and 20th editions. Method 7500–U C Fluorometric Uranium is only in the 17th Edition, and 7500–U C Alpha spectrometry is only in the 18th, 19th and 20th editions. Method 7120 is only in the 19th and 20th editions. Methods 302, 303, 304, 305 and 306 are only in the 13th edition. Method 7110 B–00, 7110 C–00, 7500–Ra B–01, 7500–Ra C–01, 7500–Ra D–01, 7500–U B–00, 7500–U C–00, 7500–I B–00, 7500–I C–00, 7500–I D–00, 7120–97, 7500–Sr B–01, and 7500–3H B–00 are available on-line at *http://www.standardmethods.org.*

[6] *Annual Book of ASTM Standards,* Vol. 11.02, 2002 ; American Society for Testing and Materials; any year containing the cited version of the method may be used. Copies may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[7] "Methods for Determination of Radioactive Substances in Water and Fluvial Sediments,'' Chapter A5 in Book 5 of *Techniques of Water-Resources Investigations of the United States Geological Survey,* 1977. Available at U.S. Geological Survey (USGS) Information Services, Box 25286, Federal Center, Denver, CO 80225–0425.

[8] "EML Procedures Manual,'' 28th (1997) or 27th (1990) Editions, Volume 1; either edition may be used. In the 27th Edition Method Ra–04 is listed as Ra–05 and Method Ga–01–R is listed as Sect. 4.5.4.3. Available at the Environmental Measurements Laboratory, U.S. Department of Energy (DOE), 376 Hudson Street, New York, NY 10014–3621.

[9] "Determination of Ra–226 and Ra–228 (Ra–02),'' January 1980, Revised June 1982. Available at Radiological Sciences Institute for Laboratories and Research, New York State Department of Health, Empire State Plaza, Albany, NY 12201.

[10] "Determination of Radium 228 in Drinking Water,'' August 1980. Available at State of New Jersey, Department of Environmental Protection, Division of Environmental Quality, Bureau of Radiation and Inorganic Analytical Services, 9 Ewing Street, Trenton, NJ 08625.

[11] Natural uranium and thorium-230 are approved as gross alpha calibration standards for gross alpha with co-precipitation and evaporation methods; americium-241 is approved with co-precipitation methods.

[12] If uranium (U) is determined by mass, a 0.67 pCi/µg of uranium conversion factor must be used. This conversion factor is based on the 1:1 activity ratio of U–234 and U–238 that is characteristic of naturally occurring uranium.

[13] "The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors.'' Available from the Environmental Resources Center, Georgia Institute of Technology, 620 Cherry Street, Atlanta, GA 30332–0335, USA, Phone: 404–8944–3776.

* * * * *

6. Section 141.74 is amended by adding one sentence to the end of footnote 1 to the table in paragraph (a)(1); and by revising paragraph (a)(2) to read as follows:

§ 141.74 Analytical and monitoring requirements.

(a) * * *

(1) * * *

* * * * *

[1] * * * In addition, the following on-line versions may also be used: 2310 B–01, 9215 B–00, 9221 A, B, C, E–99, 9222 A, B, C, D–97, and 9223–97.

* * * * *

(2) Public water systems must measure residual disinfectant concentrations with one of the analytical methods in the following table. If approved by the State, residual disinfectant concentrations for free chlorine and combined chlorine also may be measured by using DPD colorimetric test kits. In addition States may approve the use of the ITS free chlorine test strip for the determination of free chlorine. Use of the test strips is described in Method D99–003, "Free Chlorine Species'' (HOCl- and OCl-) by Test Strip,'' [Revision 3.0, November 21, 2003], available from Industrial Test Systems, Inc., 1875 Langston St., Rock Hill SC 29730. Free and total chlorine residuals may be measured continuously by adapting a specified chlorine residual method for use with a continuous monitoring instrument provided the chemistry, accuracy, and precision remain the same. Instruments used for continuous monitoring must be calibrated with a grab sample measurement at least every five days, or with a protocol approved by the State.

Residual	Methodology	SM[1]	SM on-line[2]	Other
Free Chlorine	Amperometric Titration	4500–Cl D	4500–Cl D–00	D1253–03[3]
	DPD Ferrous Titrimetric	4500–Cl F	4500–Cl F–00	
	DPD Colorimetric	4500–Cl G	4500–Cl G–00	
	Syringaldazine (FACTS)	4500–Cl H	4500–Cl H–00	
Total Chlorine	Amperometric Titration	4500–Cl D	4500–Cl D–00	D1253–03[3]
	Amperometric Titration (low level measurement).	4500–Cl E	4500–Cl E–00	
	DPD Ferrous Titrimetric	4500–Cl F	4500–Cl F–00	
	DPD Colorimetric	4500–Cl G	4500–Cl G–00	
	Iodometric Electrode	4500–Cl I	4500–Cl I–00	
Chlorine Dioxide	Amperometric Titration	4500–ClO_2 C	4500-ClO_2 C–00	
	DPD Method	4500–ClO_2 D		
	Amperometric Titration	4500–ClO_2 E	4500–ClO_2 E–00	
	Spectrophotometric			327.0, Revision 1.0[4]
Ozone	Indigo Method	4500–O_3 B	4500–O_3 B	

[1] Except for the method for ozone residuals, the listed methods are contained in the 18th, 19th, and 20th editions of Standard Methods for the Examination of Water and Wastewater, 1992, 1995, and 1998; the cited methods published in any of these three editions may be used. The ozone method, 4500–O_3 B, is contained in the 18th, 19th, and 20th editions of Standard Methods for the Examination of Water and Wastewater, 1992, 1995, and 1998, respectively; any of these editions may be used.

[2] Standard Methods On-Line are available at http://www.standardmethods.org. The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only On-Line versions that may be used.

[3] Annual Book of ASTM Standards, Vol 11.01, 2004 of any year containing the cited version of the method.

[4] EPA Method 327.0, Revision 1.0, "Determination of Chlorine Dioxide and Chlorite Ion in Drinking Water Using Lissamine Green B and Horseradish Peroxidase with Detection of Visible Spectrophotometry," USEPA, July 2003. Available on-line at *http://www.epa.gov/safewater/methods/sourcalt.html.*

* * * * *

7. Section 141.131 is amended by revising paragraph (a)(2) and the entry for "Amperometric Detection'' in the table in paragraph (c)(1) to read as follows:

§ 141.131 Analytical requirements.

(a) * * *

(2) The following documents are incorporated by reference. The Director of the Federal Register approves this incorporation by reference in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be inspected at EPA's Drinking Water Docket, 1301 Constitution Avenue, NW., EPA West B–102, Washington, DC 20460, or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington DC. EPA Method 552.1 is in *Methods for the Determination of Organic Compounds in Drinking Water-Supplement II,* USEPA, August 1992, EPA/600/R–92/129 (available through National Information Technical Service (NTIS), PB92–207703). EPA Methods 502.2, 524.2, 551.1, and 552.2 are in *Methods for the Determination of Organic Compounds in Drinking Water-Supplement III,* USEPA, August 1995, EPA/600/R–95/131. (available through NTIS, PB95–261616). EPA Method 300.0 is in *Methods for the Determination of Inorganic Substances in Environmental Samples,* USEPA, August 1993, EPA/600/R–93/100 (available through NTIS, PB94–121811). EPA Method 300.1 is titled *USEPA Method 300.1, Determination of Inorganic Anions in Drinking Water by Ion Chromatography, Revision 1.0,* USEPA, 1997, EPA/600/R–98/118 (available through NTIS, PB98–169196); also available from: Chemical Exposure Research Branch, Microbiological & Chemical Exposure Assessment Research Division, National Exposure Research Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH 45268, Fax Number: 513–569–7757, Phone number: 513–569–7586. Standard Methods 4500–C1 D, 4500–C1 E, 4500–C1 F, 4500–C1 G, 4500–C1 H, 4500–C1 I, 4500–C1O2 D, 4500–C1O2 E, 6251 B, and 5910 B shall be followed in accordance with *Standard Methods for the Examination of Water and Wastewater, 19th Edition,* American Public Health Association, 1995; copies may be obtained from the American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. Standard Methods 5310 B, 5310 C, and 5310 D shall be followed in accordance with the *Supplement to the 19th Edition of Standard Methods for the Examination of Water and Wastewater,* American Public Health Association, 1996; copies may be obtained from the American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. Standard Method (SM) on-line are available at *http://www.standardmethods.org.* ASTM Method D 1253–86 shall be followed in accordance with the *Annual Book of ASTM Standards,* Volume 11.01, American Society for Testing and Materials, 1996 or any year containing the cited version of the method may be used. ASTM Method D 1253–03 shall be followed in accordance with the *Annual Book of ASTM Standards,* Volume 11.01, 2004 or any year containing the cited version of the method may be used. Copies may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

* * * * *

(c) * * *

(1) * * *

APPROVED METHODS FOR DISINFECTANT RESIDUAL COMPLIANCE MONITORING

Methodology	Standard Method	ASTM method	Residual measured[1]			
			Free chlorine	Combined chlorine	Total chlorine	Chlorine dioxide
Amperometric Titration	4500–Cl D	D 1253–86, 03	X	X	X	....

APPROVED METHODS FOR DISINFECTANT RESIDUAL COMPLIANCE MONITORING—Continued

Methodology	Standard Method	ASTM method	Residual measured[1]			
			Free chlorine	Combined chlorine	Total chlorine	Chlorine dioxide
*	*	*	*	*	*	*

[1]X indicates method is approved for measuring specified disinfectant residual.

* * * * *

PART 143—NATIONAL SECONDARY DRINKING WATER REGULATIONS

1. The authority citation for part 143 continues to read as follows:

Authority: 42 U.S.C. 300f *et seq.*

2. Section 143.4 is amended by revising the table in paragraph (b) to read as follows:

§ 143.4 Monitoring.

* * * * *

(b) * * *

Contaminant	EPA	ASTM[3]	SM[4] 18th and 19th ed.	SM[4] 20th ed.	SM[7] On-line	Other
1. Aluminum	200.7[2]		3120 B	3120 B	3120 B–99	
	200.8[2]		3113 B		3113 B–99	
	200.9[2]		3111 D		3111 D–99	
2. Chloride	300.01[1]	D4327–97, 03	4110 B	4110 B	4110 B–00	
	....		4500–Cl D	4500–Cl D	4500–Cl D–97	
	....	D512–89(99) B	4500–C1 B	4500–Cl B	4500–Cl B–97	
	....					D6508, Rev. 2[6]
3. Color	....		2120 B	2120 B	2120 B–01	
4. Foaming Agents	....		5540 C	5540 C	5540 C–00	
5. Iron	200.7[2]		3120 B	3120 B	3120 B–99	
	200.9[2]		3111 B		3111 B–99	
	....		3113 B		3113 B–99	
6. Manganese	200.7[2]		3120 B	3120 B	3120 B–99	
	200.8[2]		3111 B		3111 B–99	
	200.9[2]		3113 B		3113 B–99	
7. Odor	....		2150 B	2150 B	2150 B–97	
8. Silver	200.7[2]		3120 B	3120 B	3120 B–99	I–3720–85[5]
	200.8[2]		3111 B		3111 B–99	
	200.9[2]		3113 B		3113 B–99	
9. Sulfate	300.0[1]	D4327–97, 03	4110 B	4110 B	4110 B–00	
	375.2[1]		4500–SO_4^2 F	4500–SO_4^2 F		
	....		4500–SO_4^{2-} C, D	4500–$SO4^{2-}$		
	....	D516–90, 02	4500–SO_4^{2-} E	4500–SO_4^{2-} E		
	....					
10. Total Dissolved Solids	....		2540 C	2540 C	2540 C–97	D6508, Rev. 2[6]
11. Zinc	200.7[2]		3120 B	3120 B	3120 B–99	
	200.8[2]		3111 B		3111 B–99	

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of the following documents 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 below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, EPA West, 1301 Constitution Avenue, NW., Room B102, Washington, DC (Telephone: 202–566–2426); or at the Office of Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC 20408.

[1] "Methods for the Determination of Inorganic Substances in Environmental Samples'', EPA/600/R–93–100, August 1993. Available at NTIS, PB94–120821.

[2] "Methods for the Determination of Metals in Environmental Samples—Supplement I'', EPA/600/R–94–111, May 1994. Available at NTIS, PB 95–125472.

[3] *Annual Book of ASTM Standards,* 1994, 1996, or 1999, Vols. 11.01 and 11.02, American Society for Testing and Materials; any year containing the cited version of the method may be used. Copies may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[4] *Standard Methods for the Examination of Water and Wastewater,* 18th edition (1992), 19th edition (1995), or 20th edition (1998). American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. The cited methods published in any of these three editions may be used, except that the versions of 3111 B, 3111 D, and 3113 B in the 20th edition may not be used.

[5] Method I–3720–85, *Techniques of Water Resources Investigation of the U.S. Geological Survey,* Book 5, Chapter A–1, 3rd ed., 1989; Available from Information Services, U.S. Geological Survey, Federal Center, Box 25286, Denver, CO 80225–0425.

[6] 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, 508/482–2131 (Office), 508/482–3625 (FAX).

[7] Standard Methods On-line are available at *http://www.standardmethods.org.*

PART 403—GENERAL PRETREATMENT REGULATIONS FOR EXISTING AND NEW SOURCES OF POLLUTION

1. The authority citation for part 403 continues to read as follows:

Authority: 33 U.S.C. 1251 *et seq.*

2. Section 403.12 is amended by removing the first sentence of paragraph (b)(5)(iii) and adding in its place four new sentences to read as follows:

§ 403.12 Reporting requirements for POTW's and industrial users.

* * * * *

(b) * * *

(5) * * *

(iii) Grab samples must be used for pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, sulfide, fecal coliform, fecal streptococcus, and volatile organics, unless specified otherwise at 40 CFR part 136. For all other pollutants, a 24-hour composite sample, using a minimum of four (4) grab samples, must be obtained through flow-proportional composite sampling techniques where feasible and unless specified otherwise at 40 CFR part 136. Results of analyses of individual grab samples for any parameter may be averaged to form the daily average. Grab samples that are not required to be analyzed immediately (see Table II at 40 CFR 136.3(e)) may be composited in the laboratory, provided that container, preservation, and holding time requirements are met (see Table II at 40 CFR 136.3(e)) and that sample integrity is not compromised by compositing. * * *

* * * * *

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

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

Authority: Sections 301, 304, 306, 307, 308, 402, and 501 of the 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).

2. Section 430.02 is amended by adding paragraph (g) to read as follows:

§ 430.02 Monitoring requirements.

* * * * *

(g) Analyst may use NCASI Method CP–86.07, “Chlorinated Phenolics in Water by *In situ* Acetylation and GC/MS Determination’’ (January 2002) for determination of certain chlorinated phenols, chlorinated guaiacols, chlorinated catechols, chlorinated benzaldehydes (*i.e.*, vanillins and syringaldehydes), and trichlorsyringol (analytes specified in the method) in bleach plant filtrate as an alternative to EPA Method 1653. NCASI Method CP–86.07 is available from the Publications Coordinator, NCASI, P.O. Box 13318, Research Triangle Park, NC 27709–3318. Phone 919–588–1987.

PART 455—PESTICIDE CHEMICALS

1. The authority citation for part 455 continues to read as follows:

Authority: Secs. 301, 304, 306, 307, and 501, Pub. L. 92–500, 86 Stat. 816, Pub. L. 95–217, 91 Stat. 156, and Pub. L. 100–4 (33 U.S.C. 1311, 1314, 1316, 1317, 1361).

2. Section 455.50 is revised to read as follows:

§ 455.50 Identification of test procedures.

The pesticide active ingredients to which this section applies and for which effluent limitations guidelines and standards are specified in this part are named, together with the Chemical Abstracts Service (CAS) number (provided to assist in identifying the pesticide active ingredient only) and analytical method(s) designation(s) in Table IG at 40 CFR 136.3(a). Except as provided in 40 CFR 136.5, the discharge parameter values required under the Clean Water Act must be determined by one of the analytical methods cited and described in Table IG at 40 CFR 136.3(a). Pesticide manufacturers may not use the analytical method cited in Table 1B, Table 1C, or Table 1D of 40 CFR 136.3(a) to make these determinations (except where the method cited in those tables is identical to the method specified in Table IG at 40 CFR 136.3(a)). The full texts of the analytical methods cited in Table IG at 40 CFR 136.3(a) are contained in the *Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I,* EPA 821–R–93–010A (August 1993 Revision I) and *Volume II,* EPA 821–R–93–010B (August 1993) (the “Compendium’’). Each pesticide chemical manufacturer that is required to determine discharge parameter values under this part using one of the analytical methods cited in Table IG at 40 CFR 136.3(a) must request in writing a copy of the Compendium from the permit authority or local control authority (as applicable) prior to determining such discharge parameter values, unless the manufacturer already has a copy.

Table 7 to Part 455 [Removed and Reserved]

3. Table 7 to part 455 is removed and reserved.

PART 465—COIL COATING POINT SOURCE CATEGORY

1. The authority citation for part 465 continues to read as follows:

Authority: Secs. 301, 304 (b), (c), (e), and (g), 306 (b) and (c), 307 (b) and (c), and 501 of the Clean Water Act (the Federal Water Pollution Control Act of 1972, as amended by the 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.

2. Section 465.03 is amended by revising paragraph (c) to read as follows:

§ 465.03 Monitoring and reporting requirements.

* * * * *

(c) The analytical method required for determination of petroleum hydrocarbons (non-polar material) is given under the listing for “oil and grease’’ at 40 CFR 136.3(a), Table IB and must be used after December 31, 2005.

* * * * *

[FR Doc. 04–6427 Filed 4–5–04; 8:45 am]

BILLING CODE 6560–50–P